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HMV: ANALYSIS OF DOSING FORMULATIONS USED IN ACUTE

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SUB-ACUTE AND SUB-CHRONIC (U) INDIAN RESEARCH

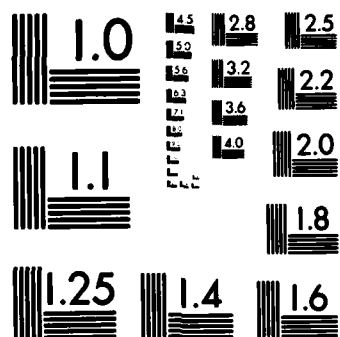
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Abstract

HMX can be detected in dosing solutions and in diets prepared for toxicity studies by reverse phase HPLC.

Dietary preparations were stable for at least 21 days when stored in ambient conditions in the dark.

Almost all the dosing solutions and diets prepared for a range of studies were shown to contain dose to the desired concentrations of HMX. There was, however, the possibility of an interchange between 2 groups on one occasion on the 13 week rat study.

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Sub-acute and Sub-chronic Toxicity Studies

Final Report by:

M.S. Henderson

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Department of the Army position unless so designated by other authorised
documents.

FOREWORD

"I, the undersigned, hereby declare that this work was performed under my supervision, according to the procedures herein described and that this report represents a true and accurate record of the results obtained."

A. B. Wilson

A.B. Wilson, B.V.Sc., M.R.C.V.S.,
D.A.B.T.
Principal Investigator



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QUALITY ASSURANCE AUTHENTICATION

The execution of this type of short-term study is not individually inspected. The processes involved are inspected at intervals according to a pre-determined schedule.

This report has been audited by IRI Quality Assurance Personnel according to the appropriate Standard Operating Procedure and is considered to describe the methods and procedures used in the study. The reported results accurately reflect the original data of the study.

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Signed: Andrew Waddell
(Quality Assurance Manager)

Date: 3rd March 1986.

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SUMMARY

An analytical method for HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine) in dosing suspensions has been developed. Aliquots of the formulation are dissolved in acetonitrile and diluted until a solution is obtained whose ultraviolet absorbance at 228 nm can be recorded.

Ten suspensions of HMX in distilled water were prepared in order to assess the accuracy of the preparation procedure and the homogeneity of the formulation. In all samples the results were within $\pm 10\%$ of the target values and were considered satisfactory.

The method was used to analyse the samples prepared during studies to evaluate the acute toxicological effects of HMX. The results are reported and, as they were within $\pm 10\%$ of the target values, were considered satisfactory.

As part of the study concerning the toxicology of HMX following dietary administration to rats and mice it was necessary to formulate HMX in diet and analyse the formulations in order to assess both the accuracy of preparation and their homogeneity. The stability of the formulated test substance on storage for up to 21 days was also assessed.

The principal findings were as follows:

1. HMX can be readily determined in diets using a procedure based on reverse phase high performance liquid chromatography (HPLC) with spectrophotometric detection. Tetryl (tetranitromethylaniline) is used as internal standard.
2. The accuracy of dietary preparation (defined as % mean found/target concentration) and the homogeneity of the diet (defined as co-efficient of variation at each of the test concentrations) were as indicated below:

Test Sample Concentration (ppm)	Accuracy (% Recovery)	Co-efficient of Variation n = 10
1,250	94.2	6.8
10,000	102.0	5.4
25,000	99.3	2.5

3. Re-analysis of the same samples after storage in darkness at ambient temperature for 21 days showed no significant degradation of HMX, i.e. values for re-assay after storage are within 2σ of the initial non-stored values.
4. Re-analysis of the same samples after storage at 40°C for 14 days showed no significant degradation of HMX.

Prior to commencement of the main 90 day subchronic studies, 14 day dose range finding studies were undertaken. As part of the latter, it was necessary to carry out routine chemical analysis of HMX in the formulated diets. Accordingly aliquots were collected and analysed at commencement and on Day 8 of the study programme.

The results of the analyses were considered acceptable since other than Group 3♀ mixed 23 October 1980 the mean values were within $\pm 10\%$ of the target value.

During the 90 day studies and the 14 day rat study aliquots of diet for consumption by animal dose groups were analysed at commencement and at Weeks 1, 2, 3, 6, 9 and at termination of the study. The results are presented in Tables 28-41.

The results are generally ($>90\%$) acceptable since the mean values were within 10% of the nominal values.

Two exceptions to this were found during the analysis of the formulations for the rat study and it is suggested on one occasion (15 December 1980) the Group 5♀ diet may have been sampled twice and on the other (29 December 1980) the diets had been interchanged although it is not possible to state whether this occurred during the formulation or the sampling of the diet.

INTRODUCTION

The U.S. Army have undertaken a programme of toxicity testing of HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine) following both dietary and oral (by gavage) administration to rats and mice. In addition, one study was concerned with the establishment of percutaneous toxicity of HMX when administered to rabbits. As an essential part of these studies it was necessary to optimise the procedure for the preparation of HMX in suspension in liquid vehicles. There was also a requirement to analyse the suspensions prior to and during the dosing phases of the study in order to assess both the homogeneity and the accuracy of the preparation of doses.

As an essential part of the dietary studies it was necessary to optimise the procedures for the preparation of the diet, to analyse the dietary mixes and to assess the stability of HMX in dietary formulations after storage.

The analytical experiments described in this report were performed at the Inveresk Gate laboratories of Inveresk Research International Limited, Musselburgh, Scotland between July 1980 and April 1981.

The formulation and sampling of the diets and the preparation of the dosing suspensions were carried out in the dispensary at the Elphinstone Research Centre.

All data generated and recorded during this study will be stored in the Scientific Archives of Inveresk Research International Limited.

GENERAL EXPERIMENTAL PROCEDURES

Materials

HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine) and RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine) were supplied from the Royal Ordnance Factory, Bridgewater, England as white suspensions containing approximately 20% by weight of water. The dry materials were obtained by drying to constant weight in a water heated oven.

Tetryl (trinitrophenylmethylnitramine) for use as internal standard was supplied by the Ministry of Defence, Waltham Abbey, England from wartime stock.

DNB (1,3-dinitrobenzene) (organic analytical standard grade) was supplied by BDH Limited, Poole, Dorset, England.

SEX (octahydro-1-acetyl-3,5,7-trinitro-1,3,5,7-tetrazocine) and TAX (hexahydro-1-acetyl-3,5-dinitro-1,3,5-triazocine) were supplied by the U.S. Army (Holston Defence Corporation).

Acetonitrile (of HPLC or "S" grade) was purchased from Rathburn Chemicals Limited, Walkerburn, Scotland.

Moisture Content of Supplied HMX

Approximately 1 g of the supplied HMX was weighed accurately into a glass scintillation vial and placed in a water-jacketted oven at 90°C for several hours. The vial was removed, placed in a desiccator for 30 min to cool and then reweighed. This process was continued until the vial reached constant weight. The experiment was performed in triplicate and the mean value used. It was found that samples could be dried to constant weight at 90°C in 3½-4 h.

Where the dry material was used for the preparation of formulations it was assumed to be fully dry when no further loss in weight was detected. No further calculation of water content was made for these samples.

EXPERIMENTAL PROCEDURES

SECTION 1: Analysis of HMX Formulations Prepared During the Acute Toxicological Studies

Measurement of the Extinction Co-efficient for HMX

Five solutions of HMX in acetonitrile were prepared and their ultraviolet spectra recorded (see Figure 1 for typical spectrum).

The data are presented below.

Initial Concentration mg.100 ml ⁻¹	Concentration After Dilution mg.100 ml ⁻¹	ΔA	ϵ	Mean Value
30.4	1.52	1.07	20837	21346 + 454 - 454
29.1	1.455	1.07	21768	
24.7	1.235	0.88	21091	
21.7	1.085	0.795	21688	

Calculation:

$$\epsilon = \frac{\Delta A \times MW \times 10^2}{C}$$

where ϵ = Extinction co-efficient for HMX

ΔA = Absorbance of solution at 228 nm

C = Concentration of measured solution (mg.100 ml⁻¹)

MW = Molecular weight

Final Analytical Methodology for the Analysis of HMX in Suspensions

The supplied formulation was mixed thoroughly on a Vortex mixer and a 1 ml aliquot of the suspension transferred to a 100 ml volumetric flask. This was made to the mark with acetonitrile (HPLC grade). For complete dissolution of the HMX it was necessary to shake thoroughly for at least 1 min or keep in an ultrasonic bath. A suitable aliquot (V.ml) was removed and transferred to a 100 ml or 250 ml volumetric flask and made to the mark with acetonitrile (S grade). The ultraviolet spectrum was recorded using as reference a solution prepared as above from 1 ml of 0.5% low-viscosity carboxymethylcellulose (CMC).

The results are tabulated (Tables 8-10).

Calculation:

$$\text{Concentration (mg.ml}^{-1}\text{)} = \frac{\Delta A \times d \times 296 \times 10^4}{W \times V \times \epsilon}$$

where ΔA = Absorbance at 228 nm

d = Density of suspension

W = Weight of aliquot

V = Volume removed for second dilution

ϵ = Extinction co-efficient for HMX (21346)

For 1% high-viscosity CMC:

$$\text{Concentration (mg.ml}^{-1}\text{)} = \frac{\Delta A \times 172.5}{W \times V}$$

For physiological saline:

$$\text{Concentration (mg.ml}^{-1}\text{)} = \frac{\Delta A \times 156.1}{W \times V}$$

It was also necessary to calculate the density of HMX as a suspension in the 2 vehicles. Aliquots (1 ml) of a 60% w/w suspension were removed and transferred to preweighed 1 ml volumetric flasks. The flasks were reweighed and the density calculated. The results are tabulated (Table 11).

The sampling schedules relating test sampling to animal dosing are shown below (Tables 1-2).

SECTION 2: Analysis of HMX in Dietary Formulations Analytical Method Development

Preliminary HPLC of HMX and Related Explosives

Solvent 1: Methanol:Water (30:70 v/v)

Solvent 2: Acetonitrile:Water (20:30 v/v)

System A

Column: 100 x 5 mm stainless steel packed with Hypersil ODS
Solvent: 1
Flow: 1 ml.min⁻¹
Wavelength: 228 nm
Detector: Pye LCUV or LC3UV
Range: 0.32 AUFS
Pump: Altex 110A
Recorder: 10 mV
Chart Speed: 600 mm.h⁻¹

The chromatograms obtained are shown in Figure 2.

System B

Column: 100 x 5 mm stainless steel packed with Hypersil ODS
Solvent: 2

The chromatograms obtained are shown in Figures 3-6.

System C

Column: 250 x 5 mm stainless steel packed with LiChrosorb RP 2
Solvent: 2
Flow: 2 ml.min⁻¹

System D

Column: 250 x 5 mm stainless steel packed with LiChrosorb RP 8
Solvent: 2
Flow: 2 ml.min⁻¹

Using conditions described under System A it was possible to separate the compounds shown (Figure 2) but it was not always possible to obtain single peaks due to solvent effects caused by the poor solubility of HMX in aqueous methanol solutions. For this reason it was decided that the preliminary HPLC experiments would be carried out using conditions described under System B. As seen from Figure 5 this system does not separate HMX from RDX or SEX from TAX, but does permit separation of HMX from tetryl which was used as internal standard (Figure 6).

During the course of the study tetryl became unavailable and 1,3-dinitrobenzene was used as internal standard (Figure 7).

Preliminary Analysis of HMX in Diet

(a) Experimental Laboratory Preparation of Diet

To 100 g of BP diet (for typical analysis see Appendix 1) was added a solution of HMX (50 mg) in acetonitrile (10 ml). After addition of 200 ml of acetonitrile the mixture was shaken mechanically for 5 min and then evaporated on a Buchi rotary evaporator for 5 h at 50°C. This gave a diet containing 500 ppm HMX.

(b) Method of Analysis

3 x 10 g samples of the diet were weighed into clear 8 oz glass jars. To each sample was added 1.5 ml of internal standard solution (38.3 mg tetryl in 10 ml acetonitrile) and acetonitrile (50 ml) as extracting solvent. The bottles were shaken mechanically for 1 h and the contents allowed to settle. An aliquot of the supernatant (10 μ l) was taken for analysis by HPLC using conditions described under System B. The peak height ratios were compared with those from extracts of diet to which known amounts of HMX and tetryl were added. The mean value obtained was 520 ± 12 ppm (relative error 4%). Comparison of peak heights obtained from extracted HMX and from known concentrations of HMX indicated that complete extraction had taken place.

Purity of HMX

Using the HPLC conditions described under System A in which HMX can be distinguished from RDX it was calculated that the supplied material contained a maximum of 0.4% RDX (Figure 8).

Final Analytical Procedure for the Determination of HMX in Diets

Approximately 5 g of the supplied diet was weighed accurately into an 8 oz clear glass jar. To the sample was added 1 ml of internal standard solution (tetryl or 1,3-dinitrobenzene in acetonitrile) and 50 ml of acetonitrile as extracting solvent. The mixture was shaken mechanically for 1 h, and the contents left to settle, with centrifugation (15 min, 3,000 r.p.m.) if necessary. An appropriate aliquot (5-50 μ l) was taken for analysis by HPLC.

Standard solutions of HMX were prepared by adding a known amount of HMX (equivalent to that of the groups being analysed) to a sample of untreated diet. To these were added internal standard solution and extracting solvent as described above. The experimental details and the results obtained from the analyses are presented in Tables 12-14.

It should be noted that the HMX used for the preparation of the calibration standards and quality control samples was from the same dried batch as that used for the preparation of the bulk diet.

Quality control samples were prepared by an independent operator at a level unknown to the analyst and included in each batch of samples processed. Standards and samples were extracted and analysed concurrently.

HPLC Conditions

Column: 100 x 5 mm stainless steel packed with Hypersil ODS
Solvent: Acetonitrile:Water (2:3 v/v)
Flow Rate: 1 ml.min⁻¹
Wavelength: 228 nm
Equipment: Hewlett Packard 1084B liquid chromatograph with a variable wavelength ultraviolet detector and an autosampler. This instrument integrates the output from the detector and calculates peak areas. When calibrated and appropriately programmed it calculates the concentration of HMX in a sample from the peak area ratios and the sample weight. The information is printed out after each chromatogram.

This method was used for the analysis of HMX in all samples of diet prepared for animal consumption during the toxicology studies.

SECTION 3: Analysis of HMX in Dietary Formulations During 14 Day and 90 Day Toxicity Testing in Rats and Mice

Preparation of Dietary Formulations

The formulations were prepared by mixing the correct amount of dry test material with the animal feedstuff in plastic drums on a Winkworth Change Drum Tumble Mixer for 20 min. Aliquots for analysis (3 x 10 g) were removed from three places through the mix.

Analytical Procedure for the Determination of HMX in Diets

The procedure used for the analysis of diets prepared for animal consumption during the toxicology studies was that described on page 9, Final Analytical Procedure for the Determination of HMX in Diets.

Sampling Schedules

Aliquots of diet (3 x 10 g) for consumption by animal dose groups 1-5♂ and 1-5♀ were analysed at commencement and on Day 8 of the 14 day study.

For the 90 day studies aliquots of diet for animal dose groups 1-6♂ and 1-6♀ were analysed at commencement and at Weeks 1,2,3,6,9 and termination of the study.

RESULTS AND DISCUSSION

SECTION 1: Analysis of HMX Formulations Prepared During the Acute Toxicological Studies.

Method Development

Ten suspensions of HMX in distilled water were supplied for analysis in order to assess the accuracy of the preparation procedures and the homogeneity of the suspensions. Each sample was analysed in duplicate and the results are shown in Table 3. In all samples the results were within $\pm 10\%$ of the target values and were considered satisfactory.

Analysis of HMX in Suspensions for the Range Finding and Main Acute Toxicology Studies in Rats and Mice

The formulations for the range finding studies were prepared in 0.5% low viscosity carboxymethylcellulose (CMC). Aliquots for analysis were removed at intervals during the dosing of each group of animals. Tables 4-7 show the results obtained. In all samples the results were within $\pm 10\%$ of the target values and were considered satisfactory.

Analysis of HMX in Suspensions During Acute Dermal Toxicity Testing in Rabbits

Samples were prepared in 1% high-viscosity CMC and physiological saline. The analysis were performed in duplicate and the results are shown in Tables 8-10.

SECTION 2: Analysis of HMX in Dietary Formulations. Analytical Method Development.

Optimisation of Dietary Preparation Procedures

Bulk samples of HMX were supplied wetted with approximately 20% water. Since it was not considered practicable to use the wetted material in diet preparations, the material was dried to constant weight prior to formulation. The weighed quantity of test material was then mixed in plastic drums on a Winkworth Change Drum Tumble Mixer for 20 min.

Three stock diets containing HMX at concentrations of 1,250, 10,000 and 25,000 ppm were prepared and analysed to assess both the accuracy of the preparation procedure and the homogeneity of the mix. Ten samples from each batch were analysed using the method described above and the results are shown in Tables 12-14. Since the values found were within $\pm 10\%$ of the theoretical value it was considered that the method of preparation was satisfactory.

Stability of HMX in Diets

Since it was necessary to provide evidence for the stability of the test compound for the lifetime of the formulation prior to commencement of the feeding trials the following stability tests were undertaken. Samples of

formulated diets were subjected to accelerated ageing by storage in an environmental cabinet for 14 days at 40°C. Further samples were stored at room temperature in darkness for 21 days.

Analysis of the stored samples gave the results shown in Tables 15-20. Table 21 summarises the results obtained and compares them with those from freshly formulated diet. All are well within the values of $+ 2 \sigma$ of the initial non-stored values and were regarded as acceptable. There was no evidence that significant degradation had occurred during the 21 days test period.

SECTION 3: Analysis of HMX in Dietary Formulations During 14 Day and 90 Day Toxicity Testing in Rats and Mice.

14 Day Studies

The results obtained are presented in Tables 22-23 (the rat study) and Tables 25-26 (the mouse study). The results were considered satisfactory since all but one gave a mean value which fell within $+ 10\%$ of the target value. The exception (Group 3♀ in the 14 day rat study mixed 23 October 1980) was also considered acceptable (mean value 10.6%).

90 Day Studies

The results are tabulated (Tables 28-41). The concentrations quoted for the standard and quality control samples are expressed in mg while those of the formulated diet extracts are in ppm.

The formulated diets were generally considered acceptable since the mean values fell within $+ 2 \sigma$ of the nominal concentration (σ being arbitrarily assigned as 5%). Where the nominal concentration was less than 50 ppm the results are considered acceptable where the mean value fell within the range of ± 5 ppm of the nominal value.

Several exceptions to the above were found, the most notable being the diets formulated on 15 December 1980 and 29 December 1980 for the 13 week rat study. On the former occasion the Group 6♀ diet on analysis gave a result of 4882 ppm rather than 12577 ppm. This value (4882 ppm) is not significantly different from that of 5019 ppm obtained from the Group 5♀ sample (nominal concentration 5151 ppm). The dispensary weighing records are consistent with the preparation of a 12577 ppm diet and it is suggested that the Group 5♀ diet may have been sampled twice. On the latter occasion, the dispensary weighing records are consistent with the preparation of the required diets at 467 ppm and 1062 ppm. The analytical results bear this out except that the analytical sample of diet labelled 467 ppm gave a result of 896 ppm while that labelled 1062 ppm diet gave 548 ppm, suggesting that the samples have been interchanged. It is not possible to state whether this happened during the formulation or the sampling stages of the procedures.

There were also several groups (detailed in tables) where the results were outwith the defined limits of acceptability which when re-analysed gave results which were considered satisfactory.

CONCLUSION

SECTION 1: Analysis of HMX Formulations Prepared During the Acute Toxicological Studies.

By using a simple analysis based on ultraviolet spectroscopy it was possible to analyse HMX when formulated as a suspension in distilled water, carboxymethylcellulose or physiological saline.

The test suspensions analysed were, in all but 2 samples, found to be acceptable since the mean values were within $\pm 2 \sigma$ of the target value where σ is arbitrarily assigned as 5%. The 2 samples which were outwith this 10% value were 10.1 and 10.5% and were also regarded as acceptable.

SECTION 2: Analysis of HMX in Dietary Formulations. Analytical Method Development.

HMX can be readily determined in diets using a procedure based on reverse phase high performance liquid chromatography with spectrophotometric detection. The accuracy of dietary preparation was considered acceptable since analysis of 3 different batches gave mean values which were within $\pm 10\%$ of the theoretical value.

HMX formulated in diet is not degraded on storage either at 40°C for 14 days or at ambient for 21 days.

SECTION 3: Analysis of HMX in Dietary Formulations During 14 Day and 90 Day Toxicity Testing in Rats and Mice.

The test diets analysed were generally (>90%) found to be acceptable since the mean values were within $\pm 2 \sigma$ where σ is arbitrarily assigned as 5% (IRI SOP/ACH/106). Where the nominal value is less than 50 ppm the results are acceptable if the mean value is within ± 5 ppm.

Two exceptions to this were found during the analysis of the formulations for the rat study and it is suggested that on one occasion (15 December 1980) the Group 5♀ diet may have been sampled twice and on the other (29 December 1980) the diets had been interchanged although it is not possible to state whether this occurred during the formulation or the sampling of the diet (see Tables 28 and 30).

TABLE 1

Analysis of HMX in Dosing Suspensions During Percutaneous
Toxicity Testing in Rabbits

Sampling Schedule: 1

Vehicle: Physiological saline

Sample 1 taken

2 rabbits dosed (1 ml.kg^{-1})

Sample 2 taken

2 rabbits dosed (3 ml.kg^{-1})

Sample 3 taken

2 rabbits dosed (5 ml.kg^{-1})

Sample 4 taken

Vehicle: 1% High viscosity CMC

Sample 5 taken

2 rabbits dosed (0.5 ml.kg^{-1})

Sample 6 taken

2 rabbits dosed (1 ml.kg^{-1})

Sample 7 taken

2 rabbits dosed (3 ml.kg^{-1})

Sample 8 taken

2 rabbits dosed (5 ml.kg^{-1})

TABLE 2

Analysis of HMX in Dosing Suspensions During Percutaneous
Toxicity Testing in Rabbits

Sampling Schedule: 2 (Non-abraded groups)

Sample 1 taken

Group 14, 200-203♂ dosed

Sample 2 taken

Group 14, 204-207♀ dosed

Sample 3 taken

Group 16, 216-219♂ dosed

Sample 4 taken

Group 16, 220-223♀ dosed

Sample 5 taken

Group 18, 232-235♂ dosed

Sample 6 taken

Group 18, 236-239♀ dosed

Sample 7 taken

Group 20, 248-251♂ dosed

Sample 8 taken

Group 20, 252-255♀ dosed

Sample 9 taken

TABLE 2 (continued)Sampling Schedule: 3 (Abraded groups)

Sample 10 taken

Group 15, 208-211♂ dosed

Sample 11 taken

Group 15, 212-215♀ dosed

Sample 12 taken

Group 17, 224-227♂ dosed

Sample 13 taken

Group 17, 228-231♀ dosed

Sample 14 taken

Group 19, 240-243♂ dosed

Sample 15 taken

Group 19, 244-247♀ dosed

Sample 16 taken

Group 21, 256-259♂ dosed

Sample 17 taken

Group 21, 260-263♀ dosed

Sample 18 taken

TABLE 3

Analysis of HMX In Dosing Suspensions In Distilled Water (Method Development)

Nominal Concen- tration (Wet) (mg.ml ⁻¹)	Nominal Concen- tration (Dry) (mg.ml ⁻¹)	Sample No.	ΔA	V (ml)	Concen- tration Found (mg.ml ⁻¹)	Mean Concen- tration (mg.ml ⁻¹)	Deviation from Nominal (Dry) (mg.ml ⁻¹)	% Deviation from Nominal
50.6	40.5	A	0.93	3.0	43.0	43.9	3.4	8.4
		B	0.97	3.0	44.8			
100.6	80.5	A	0.78	1.5	72.1	76.8	3.7	4.6
		B	0.88	1.5	81.4			
199.9	159.9	A	1.255	1.0	174.0	170.9	11.0	6.9
		B	1.21	1.0	167.8			
302.4	241.9	A	0.97	0.5	269.0	255.2	13.3	5.5
		B	0.87	0.5	241.3			
699.6	559.7	A	0.85	0.5	589.3	617.1 [†]	57.4	10.2
		B	0.93	0.5	644.8			

Date of Formulation: 2 July 1980

[†] = Second dilution = 250 ml

Date of Analysis: 3 July 1980

TABLE 3 (continued)

Nominal Concen- tration (Wet) (mg.ml ⁻¹)	Nominal Concen- tration (Dry) (mg.ml ⁻¹)	Sample No.	ΔA	V (ml)	Concen- tration Found (mg.ml ⁻¹)	Mean Concen- tration (mg.ml ⁻¹)	Deviation from Nominal (Dry) (mg.ml ⁻¹)	% Deviation from Nominal
49.8	39.8	6 A	0.84	3.0	38.8	38.4	1.4	3.5
		B	0.82	3.0	37.9			
100.0	80.0	7 A	0.93	1.5	86.0	85.6	5.6	7.0
		B	0.92	1.5	85.1			
250.0	200.0	8 A	1.53	1.0	212.2	213.6	13.6	6.8
		B	1.55	1.0	214.9			
500.0	400.0	9 A	1.57	0.5	435.4	434.1	34.1	8.5
		B	1.56	0.5	432.7			
750.0	600.0	10 A	0.94	0.5	651.7	651.7	51.7	8.6
		B	0.94	0.5	651.7			

Date of Formulation: 4 July 1980

Date of Analysis: 7 July 1980

TABLE 4

Analysis of HMX In Dosing Suspensions Formulated in Carboxymethylcellulose During
the Range Finding Study In Rats

Group	Nominal Concen- tration (Wet) (mg.ml ⁻¹)	Nominal Concen- tration (Dry) (mg.ml ⁻¹)	Sample No.	ΔA	V (ml)	Concen- tration Found (mg.ml ⁻¹)	Mean Concen- tration (mg.ml ⁻¹)	Deviation from Nominal (mg.ml ⁻¹)	% Deviation from Nominal
1	15.00	12.00	101	0.85	10.0	11.79	12.16	0.16	1.3
			102	0.94	10.0	13.03			
			103	0.84	10.0	11.65			
2	35.00	28.00	104	0.82	4.0	28.43	29.45	1.45	5.2
			105	1.06	5.0	29.40			
			106	1.10	5.0	30.51			
3	75.00	60.00	107	0.79	2.0	54.77	61.47	1.47	2.5
			108	0.90	2.0	62.40			
			109	0.97	2.0	67.25			
4	250.00	200.00	110	1.48	1.0	205.23	201.53	1.53	0.8
			111	1.36	1.0	188.59			
			112	1.52	1.0	210.78			
5	750.00	600.00	113	0.77	0.5	533.87	561.61	38.39	6.4
			114	0.82	0.5	568.54			
			115	0.84	0.5	582.41			

Date of Formulation: 9 July 1980

Date of Analysis: 11 July 1980

TABLE 5

Analysis of HMX in Dosing Suspensions Formulated in Carboxymethylcellulose During
the Range Finding Study in Mice

Group	Nominal Concen- tration (Wet) (mg.ml ⁻¹)	Nominal Concen- tration (Dry) (mg.ml ⁻¹)	Sample No.	ΔA	V (ml)	Concen- tration Found (mg.ml ⁻¹)	Mean Concen- tration (mg.ml ⁻¹)	Deviation from Nominal (mg.ml ⁻¹)	% Deviation from Nominal
1	15.00	12.00	116	0.40	4.5	12.33	13.25	1.25	10.4
			117	0.88	9.0	13.56			
			118	1.00	10.0	13.87			
2	35.00	28.00	119	1.09	5.0	30.23	29.98	1.98	7.1
			120	0.97	4.5	29.89			
			121	0.86	4.0	29.81			
3	75.00	60.00	122	0.94	2.0	65.17	61.24	1.24	2.1
			123	0.81	2.0	56.16			
			124	0.90	2.0	62.40			
4	250.00	200.00	125	1.50	1.0	208.00	205.23	5.23	2.6
			126	1.59	1.0	220.49			
			127	1.35	1.0	187.20			
5	750.00	600.00	128	0.78	0.5	540.81	584.72	15.28	2.5
			129	0.93	0.5	644.81			
			130	0.82	0.5	568.54			

Date of Formulation: 9 July 1980

Date of Analysis: 14 July 1980

TABLE 6

Analysis of HMX In Dosing Suspensions Formulated in Carboxymethylcellulose During
Acute Toxicology Studies In Rats

Group	Nominal Concen- tration (Wet) (mg.ml ⁻¹)	Nominal Concen- tration (Dry) (mg.ml ⁻¹)	Sample No.	ΔA	V (ml)	Concen- tration Found (mg.ml ⁻¹)	Mean Concen- tration (mg.ml ⁻¹)	Group Mean (mg.ml ⁻¹)	% Deviation from Nominal
1	150.00	121.05	1	1.10	1.2	127.11	127.25	127.83	5.6
			2	1.05	1.1	132.37			
			3	0.97	1.1	122.28			
			4	1.13	1.2	130.58	134.43		
			5	1.19	1.2	137.51			
			6	1.17	1.2	135.20			
			7	1.11	1.2	128.27	121.80		
			8	0.93	1.0	128.96			
			9	0.78	1.0	108.16			
2	225.00	181.58	10	0.94	0.7	186.21	183.57	184.20	1.4
			11	0.99	0.7	196.21			
			12	0.85	0.7	168.39			
			13	0.51	0.4	176.80	182.58		
			14	0.49	0.4	169.87			
			15	0.58	0.4	201.07			
			16	0.98	0.8	169.78	186.44		
			17	1.48	1.0	205.23			
			18	0.93	0.7	184.23			
3	337.50	272.36	19	0.91	0.5	252.38	262.66	272.48	0
			20	0.94	0.4	244.40			
			21	1.05	0.5	292.21			
			22	1.32	0.7	261.49	281.30		
			23	1.06	0.5	293.98			
			24	1.04	0.5	288.43			
			25	1.13	0.6	261.16	273.49		
			26	1.60	0.75	295.83			
			27	0.76	0.4	263.47			

Date of Formulation: 22 July 1980

Date of Completion of Analysis: 25 July 1980

HMX contained 19.3% water

Second Dilution = 100 ml

TABLE 6 (continued)

Group	Nominal Concen- tration (Wet) (mg.ml ⁻¹)	Nominal Concen- tration (Dry) (mg.ml ⁻¹)	Sample No.	ΔA	V (ml)	Concen- tration Found (mg.ml ⁻¹)	Mean Concen- tration (mg.ml ⁻¹)	Group Mean (mg.ml ⁻¹)	% Deviation from Nominal	
4	506.25	408.24	28	0.845	0.3	390.59	385.00	396.75	2.9	
			29	0.855	0.3	395.21				
			30	1.065	0.4	369.21				
			31	1.455	0.5	403.53	402.61			
			32	1.455	0.5	403.53				
			33	1.445	0.5	400.76	402.64			
			34	0.955	0.3	441.73				
			35	1.015	0.4	351.88				
5	759.38	612.82	36	1.495	0.5	414.62	660.99	639.42	4.3	
			37	0.96	0.2	665.62				
			38	0.92	0.2	637.88				
			39	0.98	0.2	679.48	637.88			
			40	0.87	0.2	603.21				
			41	0.97	0.2	672.55	619.39			
			42	NO SAMPLE						
			43	1.12	0.25	621.24				
			44	1.18	0.25	654.52				
			45	0.84	0.2	582.41				

TABLE 7

Analysis of HMX In Gavage Suspensions Formulated In Carboxymethylcellulose During
Acute Toxicology Studies In Mice

Group	Nominal Concen- tration (Wet) (mg.ml ⁻¹)	Nominal Concen- tration (Dry) (mg.ml ⁻¹)	Sample No.	ΔA	V (ml)	Concen- tration Found (mg.ml ⁻¹)	Mean Concen- tration (mg.ml ⁻¹)	Group Mean (mg.ml ⁻¹)	% Deviation from Nominal
1	60.00	48.00	46	0.93	2.5	51.59	46.32	45.90	4.4
			47	0.66	2.0	45.76			
			48	0.45	1.5	41.60			
			49	0.89	2.5	49.37	44.85		
			50	0.77	2.4	44.49			
			51	0.44	1.5	40.68			
			52	0.75	2.0	52.00	46.53		
			53	0.49	1.5	45.30			
			54	0.61	2.0	42.29			
2	102.00	81.6	55	0.82	1.5	75.81	73.34	79.15	3.0
			56	0.78	1.5	72.11			
			57	1.04	2.0	72.11			
			58	0.56	1.0	77.66	80.74		
			59	0.97	1.5	89.67			
			60	0.81	1.5	74.88			
			61	1.25	2.0	86.76	83.36		
			62	0.73	1.2	84.36			
			63	0.57	1.0	79.04			
3	173.4	138.72	64	0.90	1.0	124.80	124.46	126.88	8.5
			65	0.83	1.0	115.10			
			66	0.77	0.8	133.47			
			67	1.22	1.3	130.14	134.44		
			68	1.04	1.0	144.22			
			69	0.93	1.0	128.96			
			70	0.82	0.9	126.34	121.73		
			71	0.86	1.0	119.26			
			72	0.69	0.8	119.60			

Date of Formulation: 22 July 1980

Date of Completion of Analysis: 25 July 1980

HMX contained 20% water

TABLE 7 (continued)

Group	Nominal Concen- tration (Wet) (mg.ml ⁻¹)	Nominal Concen- tration (Dry) (mg.ml ⁻¹)	Sample No.	ΔA	V (ml)	Concen- tration Found (mg.ml ⁻¹)	Mean Concen- tration (mg.ml ⁻¹)	Group Mean (mg.ml ⁻¹)	% Deviation from Nominal
4	294.78	235.82	73	1.04	0.6	240.36		240.16	1.8
			74	0.97	0.55	244.56	244.84		
			75	0.99	0.55	249.61			
			76	0.70	0.4	242.27			
			77	1.26	0.7	249.61	239.90		
			78	0.82	0.5	227.42			
			79	0.53	0.3	244.98			
			80	0.51	0.3	235.74	235.74		
			81	0.49	0.3	226.49			
5	501.13	400.90	82	1.06	0.35	419.97		413.34	3.1
			83	1.02	0.35	404.12	427.89		
			84	1.16	0.35	459.59			
			85	1.31	0.40	454.14			
			86	0.56	0.20	388.28	402.84		
			87	0.66	0.25	366.09			
			88	NO SAMPLE					
			89	1.10	0.35	435.82	409.28		
			90	0.69	0.25	382.73			

TABLE 8

Analysis of HMX in Dosing Suspensions Formulated During
 Percutaneous Toxicity Testing in Rabbits
 Nominal Concentration 600 mg.ml⁻¹

Vehicle	Sample No.	Weight of HMX Suspension (g)	V (ml)	ΔA	Concentration Found (mg.ml ⁻¹)	Mean Value (mg.ml ⁻¹)	Mean Value (mg.ml ⁻¹)	% Deviation from Nominal
Physiological Saline	1	A	1.1101	0.3	1.12	525.0	537.1 + 10.1 (1.9%)	10.5
		B	0.3193*	0.65	1.46	549.1		
	2	A	1.4153	0.7†	1.34	524.1		
		B	0.0801	1.8	0.99	535.9		
	3	A	0.1278*	1.5	1.30	529.3		
		B	1.3352	0.65†	1.22	548.6		
	4	A	0.7626*	0.35†	0.74	541.0		
		B	0.6460	1.2†	1.08	543.7		
1% High Viscosity Carboxymethyl-cellulose	5	A	0.4121	0.8	1.10	575.6	539.3 + 20.2 (3.7%)	10.1
		B	0.0877	3.5	0.995	559.2		
	6	A	0.9301	0.45	1.28	527.5		
		B	0.4480	0.9	1.26	539.1		
	7	A	0.4600	0.7	0.99	530.4		
		B	0.4467	0.8	1.06	511.7		
	8	A	0.7542	0.4	0.95	543.2		
		B	0.5280	0.7	1.13	527.4		

Date of Formulation: 11 August 1980

Date of Analysis: 14 August 1980

* = Initial volume 50 ml

† = Final volume 250 ml

TABLE 9

Analysis of HMX in Dosing Suspensions Formulated During Percutaneous Toxicity
Testing in Rabbits (Non-abraded Groups)
Nominal Concentration 600 mg.ml⁻¹ (60%)

Sample No.	Weight of HMX Suspension (g)	V (ml)	ΔA	Concentration Found (mg.ml ⁻¹)	Concentration Found (mg.ml ⁻¹)	Mean Value (%)	% Deviation from Nominal
1	0.7670	0.25	0.735	661.2	66.1		
2	0.8020	0.3	0.96	688.3	68.8		
3	0.8524	0.5	1.50	607.1	60.7		
4	0.9495	0.4	1.39	631.3	63.1	63.0	
5	0.8924	0.4	1.29	623.4	62.3	+ 3.7	5.0
6	0.6855	0.55	1.34	613.1	61.3	(6%)	
7	0.8845	0.35	1.045	582.2	58.2		
8	0.6811	0.4	0.93	588.8	58.9		
9	0.7070	0.3	0.83	675.0	67.5		

Vehicle: 1% High-viscosity carboxymethylcellulose

Date of Formulation: 14 October 1980

Date of Analysis: 15 October 1980

TABLE 10

Analysis of HMX in Dosing Suspensions Formulated During Percutaneous Toxicity
Testing in Rabbits (Abraded Groups)
Nominal Concentration 600 mg.ml⁻¹ (60%)

Sample No.	Weight of HMX Suspension (g)	V (ml)	ΔA	Concentration Found (mg.ml ⁻¹)	Concentration Found (%)	Mean Value (%)	% Deviation from Nominal
10	0.7784	0.25	0.74	656.0	65.6		
11	0.7367	0.45	1.27	660.8	66.1		
12	0.6172	0.55	1.285	653.0	65.3		
13	0.8084	0.4	1.19	634.8	63.5	64.4	
14	0.6880	0.45	1.14	635.2	63.5	+ 1.9	7.3
15	0.9135	0.3	1.1045	657.8	65.8	(2.9%)	
16	0.8206	0.35	1.085	651.7	65.2		
17	0.7328	0.4	1.02	600.3	60.0		
18	0.7868	0.4	1.18	646.8	64.7		

Vehicle: 1% High-viscosity carboxymethylcellulose

Date of Formulation: 15 October 1980

Date of Analysis: 20 October 1980

TABLE 11

Calculation of Density of HMX Suspensions

Sample No.	Vehicle	Weight Flask (g)	Weight Flask + HMX (g)	Weight HMX (g)	Density
1	1% High Viscosity Carboxymethyl-cellulose	4.9587	6.2116	1.2529	1.244
2		6.4865	7.7638	1.2773	
3		5.5393	6.7776	1.2383	
4		6.8965	8.1077	1.2112	
5		7.0406	8.2804	1.2398	
6	Physiological Saline	5.3692	6.4963	1.1271	1.126
7		5.2857	6.4068	1.1211	
8		6.9042	8.0174	1.1132	
9		6.6210	7.7641	1.1431	
10		5.6160	6.7406	1.1246	

TABLE 12

Analysis of HMX in Diets (Method Development)
Nominal Concentration 1,250 ppm

Date of Mixing: 8 September 1980

Date of Analysis: 10 September 1980

Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Peak Height Compound (mm)	Peak Height Internal Standard (mm)	Peak Height Ratio	Concen- tration Found (mg)	Concen- tration (ppm)
1	5.09	-	-	50	0	0	0		
2	5.10	1	1	50	77	56.5	1.363		
3	4.93	1	1	50	167	124	1.347		
4	4.99	1	1	50	120	87.5	1.371		
5	4.89	-	1	50	143	98	1.459	6.458	1321
6	5.25	-	1	50	114	82.5	1.382	6.117	1165
7	5.15	-	1	50	121	86	1.407	6.228	1209
8	4.99	-	1	50	97	77	1.260	5.577	1118
9	4.93	-	1	50	85	70	1.214	5.374	1090
10	5.00	-	1	50	127	98	1.296	5.737	1147
11	4.86	-	1	50	93	76	1.224	5.418	1115
12	5.09	-	1	50	80	53	1.509	6.680	1312
13	5.06	-	1	50	119	92	1.293	5.723	1131
14	5.14	-	1	50	153	113	1.354	5.993	1166

HMX solution 6.02 mg.ml⁻¹

Internal standard solution 6.16 mg.ml⁻¹

Mean value 1177 ± 80 (6.8%). Deviation of mean from nominal 73 ppm (5.8%)

TABLE 13

Analysis of HMX in Diets (Method Development)
Nominal Concentration 10,000 ppm

Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Peak Height Compound (mm)	Peak Height Internal Standard (mm)	Peak Height Ratio	Concen- tration Found (mg)	Concen- tration (ppm)
15	2.48	-	-	104	0	0	0		
16	2.51	4	1	100	148.5	80	1.856		
17	2.53	4	1	100	171.5	90.5	1.895		
18	2.50	4	1	100	67	34	1.971		
19	2.57	-	1	104	175	85.5	2.047	27.76	10800
20	2.56	-	1	104	180	100.5	1.791	24.29	9487
21	2.47	-	1	104	140.5	68	2.066	28.02	11344
22	2.50	-	1	104	165.5	90.5	1.829	24.80	9920
23	2.48	-	1	104	165	87.5	1.886	25.58	10313
24	2.57	-	1	104	156.5	79	1.981	26.86	10453
25	2.55	-	1	104	165	87.5	1.886	25.58	10030
26	2.51	-	1	104	148	80	1.850	25.09	9996
27	2.55	-	1	104	143	78	1.833	24.86	9748
28	2.49	-	1	104	161.5	89	1.815	24.61	9885

HMX solution 6.465 mg.ml⁻¹

Internal standard solution 20.36 mg.ml⁻¹

Mean value 10198 \pm 548 (5.4%). Deviation of mean from nominal 198 ppm (2.0%)

TABLE 14

Analysis of HMX in Diets (Method Development)
Nominal Concentration 25,000 ppm

Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Peak Height Compound (mm)	Peak Height Internal Standard (mm)	Peak Height Ratio	Concen- tration Found (mg)	Concen- tration (ppm)
29	2.52	-	-	110	0	0	0		
30	2.48	10	1	100	109.5	73	1.500		
31	2.49	10	1	100	151	99	1.525		
32	2.47	10	1	100	155	104	1.490		
33	2.53	-	1	110	133	89	1.494	63.97	25280
34	2.51	-	1	110	128	88	1.455	62.30	24820
35	2.51	-	1	110	113	76.5	1.477	63.24	25200
36	2.54	-	1	110	128	87.5	1.463	62.64	24660
37	2.51	-	1	110	128	87	1.471	62.98	25090
38	2.56	-	1	110	125	87	1.437	61.53	24040
39	2.49	-	1	110	133	95	1.400	59.94	24070
40	2.55	-	1	110	140	98	1.429	61.19	24000
41	2.51	-	1	110	126.5	86	1.471	62.98	25090
42	2.47	-	1	110	142	95	1.495	64.01	25910

HMX solution 6.444 mg.ml⁻¹

Internal standard solution 62.27 mg.ml⁻¹

Mean value 24816 \pm 629 (2.5%). Deviation of mean from nominal 184 ppm (0.7%)

TABLE 15

Analysis of HMX in Diets (Method Development)
 (After Storage at 40°C for 14 Days)
 Nominal Concentration 1,250 ppm

Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution (ml)	Volume Internal Standard (ml)	Volume Extracting Solvent (ml)	Concen- tration Found (ppm)
43	5.07	-	-	50	-
44	5.01	1	1	50	-
45	5.02	1	1	50	-
46	4.99	1	1	50	-
47	5.01	-	1	50	1296
48	5.00	-	1	50	1287
49	4.98	-	1	50	1259
50	4.97	-	1	50	1266
51	5.02	-	1	50	1197
52	5.00	-	1	50	1206
53	4.99	-	1	50	1272
54	4.98	-	1	50	1207
55	4.97	-	1	50	1171
56	5.00	-	1	50	1182

HMX solution 6.06 mg.ml⁻¹

Internal standard solution 6.00 mg.ml⁻¹

Mean value 1234 ± 46 (3.8%). Deviation of mean from nominal 16 ppm (1.3%)

TABLE 16

Analysis of HMX In Diets (Method Development)
 (After Storage at 40°C for 14 Days)
 Nominal Concentration 10,000 ppm

Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution (ml)	Volume Internal Standard (ml)	Volume Extracting Solvent (ml)	Concen- tration Found (ppm)
57	2.50	-	-	104	-
58	2.55	4	1	100	-
59	2.44	4	1	100	-
60	2.49	4	1	100	-
61	2.53	-	1	104	10152
62	2.51	-	1	104	10701
63	2.48	-	1	104	10315
64	2.51	-	1	104	10551
65	2.55	-	1	104	10196
66	2.52	-	1	104	9459
67	2.49	-	1	104	9769
68	2.48	-	1	104	10137
69	2.51	-	1	104	9829
70	2.50	-	1	104	10186

HMX solution 6.485 mg.ml⁻¹

Internal standard solution 20.00 mg.ml⁻¹

Mean value 10130 \pm 368 (3.6%). Deviation of mean from nominal 130 ppm (1.3%)

TABLE 17

Analysis of HMX in Diets (Method Development)
 (After Storage at 40°C for 14 Days)
 Nominal Concentration 25,000 ppm

Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution (ml)	Volume Internal Standard (ml)	Volume Extracting Solvent (ml)	Concen- tration Found (ppm)
71	2.50	-	-	110	-
72	2.54	10	1	100	-
73	2.49	10	1	100	-
74	2.50	10	1	100	-
75	2.48	-	1	110	25443
76	2.48	-	1	110	26467
77	2.49	-	1	110	25742
78	2.48	-	1	110	25354
79	2.48	-	1	110	24993
80	2.48	-	1	110	25428
81	2.52	-	1	110	25202
82	2.50	-	1	110	25238
83	2.50	-	1	110	23481
84	2.52	-	1	110	22690

HMX solution 6.494 mg.ml⁻¹

Internal standard solution 62.42 mg.ml⁻¹

Mean value 25004 \pm 1103 (4.4%). Deviation of mean from nominal 4 ppm (0%)

TABLE 18

Analysis of HMX in Diets (Method Development)
 (After Storage at Ambient for 21 Days)
 Nominal Concentration 1,250 ppm

Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution (ml)	Volume Internal Standard (ml)	Volume Extracting Solvent (ml)	Concen- tration Found (ppm)
85	4.94	-	-	50	-
86	5.02	1	1	50	-
87	5.00	1	1	50	-
88	5.02	1	1	50	-
89	5.01	-	1	50	1268
90	5.01	-	1	50	1327
91	5.04	-	1	50	1269
92	5.02	-	1	50	1380
93	5.00	-	1	50	1270
94	5.03	-	1	50	1307
95	5.00	-	1	50	1336
96	5.03	-	1	50	1311
97	5.00	-	1	50	1217
98	5.02	-	1	50	1228

HMX solution 5.94 mg.ml⁻¹

Internal standard solution 5.86 mg.ml⁻¹

Mean value 1291 \pm 50 (3.9%). Deviation of mean from nominal 41 ppm (3.3%)

TABLE 19

Analysis of HMX in Diets (Method Development)
 (After Storage at Ambient for 21 Days)
 Nominal Concentration 10,000 ppm

Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution (ml)	Volume Internal Standard (ml)	Volume Extracting Solvent (ml)	Concen- tration Found (ppm)
99	2.51	-	1	104	-
100	2.55	4	1	100	-
101	2.50	4	1	100	-
102	2.51	4	1	100	-
103	2.55	-	1	104	10740
104	2.50	-	1	104	10521
105	2.49	-	1	104	10002
106	2.54	-	1	104	10030
107	2.53	-	1	104	10568
108	2.51	-	1	104	10560
109	2.44	-	1	104	10062
110	2.51	-	1	104	10485
111	2.50	-	1	104	9807
112	2.51	-	1	104	10331

HMX solution 6.52 mg.ml⁻¹

Internal standard solution 20.01 mg.ml⁻¹

Mean value 10311 ± 312 (3.0%). Deviation of mean from nominal 311 ppm (3.1%)

TABLE 20

Analysis of HMX in Diets (Method Development)
 (After Storage at Ambient for 21 Days)
 Nominal Concentration 25,000 ppm

Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution (ml)	Volume Internal Standard (ml)	Volume Extracting Solvent (ml)	Concen- tration Found (ppm)
113	2.48	-	-	110	-
114	2.51	10	1	100	-
115	2.48	10	1	100	-
116	2.53	10	1	100	-
117	2.53	-	1	110	24905
118	2.46	-	1	110	25945
119	2.50	-	1	110	25432
120	2.49	-	1	110	27191
121	2.53	-	1	110	24044
122	2.50	-	1	110	26280
123	2.53	-	1	110	25983
124	2.50	-	1	110	24876
125	2.50	-	1	110	26241
126	2.52	-	1	110	25081

HMX solution 6.518 mg.ml^{-1}

Internal standard solution 64.31 mg.ml^{-1}

Mean value 25598 ± 907 (3.5%). Deviation of mean from nominal 598 ppm (2.4%)

TABLE 21

Analysis of HMX In Diets (Method Development)
Summary of Results

Nominal Concentration (ppm)	Concentration (ppm) Found		
	Freshly Prepared	Storage at 40°C for 14 Days	Storage at Ambient for 21 Days
1,250	1177 \pm 80	1234 \pm 46	1291 \pm 50
10,000	10198 \pm 548	10130 \pm 368	10311 \pm 312
25,000	24816 \pm 629	25004 \pm 1103	25598 \pm 907

TABLE 22

Analysis of HMX in Diets Formulated During the 14 Day Toxicity
Study in Rats (at commencement)

Date of Mixing: 16 October 1980

Date of Analysis: 27 October 1980

Nominal Concen- tration (ppm)	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Concen- tration Found (ppm)	Mean Concen- tration Found (ppm)	Deviation of Mean Concen- tration (%)
0	19♂	1	5.00	-	-	100	0	0	0
		2	5.01	-	-	100	0		
		3	5.03	-	-	100	0		
2714	2♂	4	5.00	-	1	100	2723	2744	1.1
		5	5.02	-	1	100	2842		
		6	5.00	-	1	100	2667		
2642	2♀	7	5.00	-	1	100	2744	2765	4.7
		8	5.00	-	1	100	2648		
		9	5.01	-	1	100	2903		
Standards		10	5.00	1	1	100			
		11	5.02	1	1	100			
		12	5.02	1	1	100			

HMX solution 12.60 mg.ml⁻¹Internal standard solution 12.08 mg.ml⁻¹

7650	3♂	13	3.00	-	1	104	7366	7642	0.1
		14	3.00	-	1	104	7558		
		15	3.00	-	1	104	8002		
8133	3♀	16	3.00	-	1	104	8430	8051	1.0
		17	3.00	-	1	104	7855		
		18	3.00	-	1	104	7867		
Standards		19	3.03	4	1	100			
		20	3.00	4	1	100			
		21	3.02	4	1	100			

HMX solution 6.135 mg.ml⁻¹Internal standard solution 24.05 mg.ml⁻¹

TABLE 22 (continued)

Nominal Concentration (ppm)	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Concentration Found (ppm)	Mean Concentration Found (ppm)	Deviation of Mean Concentration (%)
23550	4♂	22	2.50	-	1	110	23322	23113	1.9
		23	2.50	-	1	110	22527		
		24	2.49	-	1	110	23489		
24800	4♀	25	2.50	-	1	110	25327	25376	2.3
		26	2.50	-	1	110	25647		
		27	2.50	-	1	110	25155		
Standards		28	2.50	10	1	100			
		29	2.50	10	1	100			
		30	2.51	10	1	100			

HMX solution 6.560 mg.ml⁻¹Internal standard solution 61.465 mg.ml⁻¹

72000	5♂	31	2.50	-	1	130	67087	69955	2.8
		32	2.49	-	1	130	70922		
		33	2.50	-	1	130	71856		
72000	5♀	34	2.50	-	1	130	72017	70756	1.7
		35	2.50	-	1	130	69442		
		36	2.50	-	1	130	70809		
Standards		37	2.50	30	1	100			
		38	2.50	30	1	100			
		39	2.50	30	1	100			

HMX solution 6.075 mg.ml⁻¹Internal standard solution 179.92 mg.ml⁻¹

TABLE 23

Analysis of HMX In Diets Formulated During the 14 Day Toxicity
Study In Rats (at Day 8)

Date of Mixing: 23 October 1980

Date of Analysis: 3 November 1980

Nominal Concen- tration (ppm)	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Concen- tration Found (ppm)	Mean Concen- tration Found (ppm)	Deviation of Mean Concen- tration (%)
0	1♀	82	5.00	-	-	104	0	0	0
		83	5.01	-	-	104	0		
		84	5.01	-	-	104	0		
4995	2♀	85	5.00	-	1	104	5107	4930	1.3
		86	5.00	-	1	104	4893		
		87	5.00	-	1	104	4791		
3869	2♂	88	5.00	-	1	104	4055	3935	1.7
		89	5.00	-	1	104	3876		
		90	5.00	-	1	104	3873		
Standards		91	5.00	4	1	100			
		92	5.00	4	1	100			
		93	5.00	4	1	100			

HMX solution 5.01 mg.ml⁻¹

Internal standard solution 19.905 mg.ml⁻¹

15000	3♀	94	2.50	-	1	105	16969	16586	10.6
		95	2.51	-	1	105	16941		
		96	2.50	-	1	105	15847		
11448	3♂	97	2.50	-	1	105	10752	10785	5.8
		98	2.50	-	1	105	10933		
		99	2.51	-	1	105	10671		
Standards		100	2.50	5	1	100			
		101	2.50	5	1	100			
		102	2.50	5	1	100			

HMX solution 7.49 mg.ml⁻¹

Internal standard solution 28.04 mg.ml⁻¹

TABLE 23 (continued)

Nominal Concentration (ppm)	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Concentration Found (ppm)	Mean Concentration Found (ppm)	Deviation of Mean Concentration (%)
49000	4♀	103	2.50	-	1	120	48703	48739	0.5
		104	2.50	-	1	120	48370		
		105	2.50	-	1	120	49143		
36720	4♂	106	2.50	-	1	120	37858	36961	0.7
		107	2.50	-	1	120	36512		
		108	2.49	-	1	120	36514		
Standards		109	2.50	20	1	100			
		110	2.50	20	1	100			
		111	2.50	20	1	100			

HMX solution 5.96 mg.ml⁻¹Internal standard solution 100.06 mg.ml⁻¹

175500	5♀	112	2.00	-	4	160	175287	177482	1.1
		113	2.00	-	4	160	181643		
		114	2.00	-	4	160	175516		
Standards		115	2.00	60	4	100			
		116	2.00	60	4	100			
		117	2.00	60	4	100			

HMX solution 5.99 mg.ml⁻¹Internal standard solution 90.086 mg.ml⁻¹

120600	5♂	118	2.00	-	3	140	129395	125781	4.3
		119	2.00	-	3	140	125597		
		120	2.00	-	3	140	122352		
Standards		121	2.00	40	3	100			
		122	2.00	40	3	100			
		123	2.00	40	3	100			

HMX solution 6.004 mg.ml⁻¹Internal standard solution 79.89 mg.ml⁻¹

TABLE 24

Analysis of HMX in Diet: Results of Quality Control Sample Analyses
 [Analysed Concurrently with Diets Formulated During the 14 Day
 Toxicity Study in Rats (at Day 8)]

Group	Nominal Weight (mg)	Analytical Sample No.	Weight Found (mg)	Mean Weight (mg)	Deviation of Mean (%)
13♀ 23♀	18.52	124 125 126	18.32 18.93 20.34	19.20	3.8
33♀	25.60	127 128 129	25.35 25.52 25.80	25.56	0.2
43♀	127.40	130 131 132	126.56 126.35 123.32	125.41	1.6
5♀	413.42	133 134 135	423.58 409.84 405.43	412.95	0.1
5♂	278.35	136 137 138	260.57 281.61 269.22	270.47	2.8

TABLE 25

Analysis of HMX in Diets Formulated During the 14 Day Toxicity
Study in Mice (at commencement)

Date of Mixing: 20 October 1980

Date of Analysis: 3 November 1980

Nominal Concen- tration (ppm)	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Concen- tration Found (ppm)	Mean Concen- tration Found (ppm)	Deviation of Mean Concen- tration (%)
0	1♀	40	5.00	-	-	100	0	0	0
		41	5.01	-	-	100	0		
		42	5.02	-	-	100	0		
483	2♂	43	5.00	-	1	100	527	521	7.9
		44	5.00	-	1	100	478		
		45	5.00	-	1	100	559		
Standards		46	5.00	1	1	100			
		47	5.00	1	1	100			
		48	5.00	1	1	100			

HMX solution 2.40 mg.ml⁻¹

Internal standard solution 2.468 mg.ml⁻¹

1331	2♀	49	5.01	-	1	50	1295	1320	0.8
		50	5.00	-	1	50	1293		
		51	5.00	-	1	50	1371		
1513	3♂	52	5.02	-	1	50	1534	1547	2.2
		53	5.00	-	1	50	1515		
		54	5.00	-	1	50	1593		
Standards		55	5.00	1	1	50			
		56	5.00	1	1	50			
		57	5.00	1	1	50			

HMX solution 6.68 mg.ml⁻¹

Internal standard solution 7.032 mg.ml⁻¹

TABLE 25 (continued)

Nominal Concen- tration (ppm)	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Concen- tration Found (ppm)	Mean Concen- tration Found (ppm)	Deviation of Mean Concen- tration (%)
3325	3g	58	5.02	-	1	104	3088	3152	5.2
		59	5.00	-	1	104	3115		
		60	5.00	-	1	104	3253		
4856	4g	61	5.00	-	1	104	4833	5278	8.7
		62	4.99	-	1	104	4562		
		63	5.01	-	1	104	6439		
Standards		64	5.00	4	1	100			
		65	5.02	4	1	100			
		66	5.02	4	1	100			

HMX solution 4.992 mg.ml⁻¹Internal standard solution 20.136 mg.ml⁻¹

7926	4g	67	2.50	-	1	105	7878	8109	2.3
		68	2.50	-	1	105	7835		
		69	2.49	-	1	105	8614		
12547	5g	70	2.50	-	1	105	11852	12820	2.2
		71	2.52	-	1	105	12904		
		72	2.51	-	1	105	13705		
Standards		73	2.50	5	1	100			
		74	2.50	5	1	100			
		75	2.50	5	1	100			

HMX solution 5.008 mg.ml⁻¹Internal standard solution 24.77 mg.ml⁻¹

21078	5g	76	2.50	-	1	110	22237	22227	5.5
		77	2.51	-	1	110	22115		
		78	2.52	-	1	110	22328		
Standards		79	2.50	10	1	100			
		80	2.50	10	1	100			
		81	2.50	10	1	100			

HMX solution 5.058 mg.ml⁻¹Internal standard solution 50.184 mg.ml⁻¹

TABLE 26

Analysis of HMX in Diets Formulated During the 14 Day Toxicity
Study in Mice (at Day 8)

Date of Mixing: 27 October 1980

Date of Analysis: 3 November 1980

Nominal Concen- tration (ppm)	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Concen- tration Found (ppm)	Mean Concen- tration Found (ppm)	Deviation of Mean Concen- tration (%)
0	1♂♀	140	5.00	-	-	100	0	0	0
		141	5.00	-	-	100	0		
		142	5.00	-	-	100	0		
500	2♂	143	5.00	-	1	100	548	515	3.0
		144	5.00	-	1	100	505		
		145	5.00	-	1	100	493		
1458	2♀	146	5.00	-	1	100	1463	1521	4.3
		147	5.00	-	1	100	1547		
		148	5.00	-	1	100	1553		
Standards		149	5.00	1	1	100			
		150	5.00	1	1	100			
		151	5.00	1	1	100			

HMX solution 5.064 mg.ml⁻¹Internal standard solution 5.012 mg.ml⁻¹

2014	3♂	152	5.00	-	1	104	2026	2082	3.4
		153	5.00	-	1	104	2127		
		154	5.00	-	1	104	2093		
4556	3♀	155	5.00	-	1	104	4698	4852	6.5
		156	5.00	-	1	104	4916		
		157	5.00	-	1	104	4941		
Standards		158	5.00	4	1	100			
		159	5.00	4	1	100			
		160	5.00	4	1	100			

HMX solution 3.995 mg.ml⁻¹Internal standard solution 15.920 mg.ml⁻¹

TABLE 26 (continued)

Nominal Concen- tration (ppm)	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Concen- tration Found (ppm)	Mean Concen- tration Found (ppm)	Deviation of Mean Concen- tration (%)
11613	4♀	161	2.50	-	1	105	11221	11209	3.5
		162	2.50	-	1	105	11256		
		163	2.50	-	1	105	11151		
Standards		164	2.50	5	1	100			
		165	2.50	5	1	100			
		166	2.50	5	1	100			

HMX solution 5.996 mg.ml⁻¹Internal standard solution 29.888 mg.ml⁻¹

TABLE 27

Analysis of HMX In Diets: Results of Quality Control Sample Analyses
 [Analysed Concurrently with Diets Formulated During the 14 Day
 Toxicity Study in Mice (at Day 8)]

Group	Nominal Weight (mg)	Analytical Sample No.	Weight Found (mg)	Mean Weight (mg)	Deviation of Mean (%)
1♂ 2♂	5.34	167 168 169	5.24 5.47 5.45	5.39	0.9
3♂	17.92	170 171 172	18.03 17.78 17.59	17.80	0.4
4♀	36.03	173 174 175	34.96 35.33 34.68	35.00	2.8

TABLE 28

Analysis of HMX in Diets During 90 Day Toxicity Testing in Rats

Date of Mixing: 23 October 1980

Date of Analysis: 3 November 1980

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0	123	176	5.02	-	-	100	0	0	0
		177	4.98	-	-	100	0		
		178	5.03	-	-	100	0		
2.02	Standards	179	5.00	1	1	100			
		180	4.99	1	1	100			
		181	4.93	1	1	100			
2.13	QC	182	4.97	1	1	100	1.73	1.98	7.0
		183	5.01	1	1	100	2.10		
		184	5.00	1	1	100	2.10		
420	23	185	5.07	-	1	100	417	409	2.6
		186	5.07	-	1	100	413		
		187	4.98	-	1	100	398		
415	22	188	5.02	-	1	100	438	438	5.5*
		189	5.02	-	1	100	637		
		190	5.00	-	1	100	438		

HMX solution 2.02 mg.ml⁻¹Internal standard solution 1.97 mg.ml⁻¹QC solution of HMX 2.13 mg.ml⁻¹

* = Mean of 2 results only

TABLE 28 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
5.51	Standards	191	5.03	1	1	100			
		192	5.01	1	1	100			
		193	4.92	1	1	100			
5.20	QC	194	5.03	1	1	100	5.34	5.37	3.3
		195	5.00	1	1	100	5.50		
		196	4.95	1	1	100	5.27		
1250	33	197	4.99	-	1	100	1281	1274	1.9
		198	4.97	-	1	100	1283		
		199	4.96	-	1	100	1258		
864	32	200	5.02	-	1	100	896	877	1.5
		201	5.00	-	1	100	873		
		202	5.00	-	1	100	861		

HMX solution 5.51 mg.ml⁻¹Internal standard solution 4.94 mg.ml⁻¹QC solution of HMX 5.20 mg.ml⁻¹

TABLE 28 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
12.15	Standards	203	5.00	3	1	100			
		204	4.97	3	1	100			
		205	5.10	3	1	100			
11.91	QC	206	5.01	3	1	100	12.05	11.81	0.8
		207	4.95	3	1	100	11.98		
		208	4.99	3	1	100	11.41		
2305	4Q	209	5.03	-	1	103	2360	2309	0.2
		210	5.10	-	1	103	2290		
		211	5.10	-	1	103	2276		

HMX solution 4.05 mg.ml⁻¹Internal standard solution 12.295 mg.ml⁻¹QC solution of HMX 3.97 mg.ml⁻¹

TABLE 28 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
22.575	Standards	212	5.04	5	1	100			
		213	5.00	5	1	100			
		214	4.99	5	1	100			
22.78	QC	215	5.02	5	1	100	22.75	22.76	0
		216	5.08	5	1	100	22.42		
		217	5.00	5	1	100	23.11		
3930	43	218	5.05	-	1	105	3973	3936	0.2
		219	4.97	-	1	105	3859		
		220	4.89	-	1	105	3977		
5151	52	221	4.97	-	1	105	4918	5019	2.6
		222	5.00	-	1	105	4933		
		223	4.94	-	1	105	5205		

HMX solution 4.515 mg.ml⁻¹Internal standard solution 22.3 mg.ml⁻¹QC solution of HMX 4.555 mg.ml⁻¹

TABLE 28 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
30.07	Standards	224	5.07	6	1	100			
		225	5.02	6	1	100			
		226	5.05	6	1	100			
29.71	QC	227	5.03	6	1	100	30.63	30.27	1.8
		228	5.00	6	1	100	30.03		
		229	5.00	6	1	100	30.06		
11520	5♂	230	5.07	-	1	106	11135	11293	2.0
		231	5.00	-	1	106	11093		
		232	5.02	-	1	106	11650		
12577	6♀	233	5.02	-	1	106	4814	4882	61.2
		234	4.90	-	1	106	5004		
		235	5.00	-	1	106	4827		

HMX solution 5.012 mg.ml⁻¹Internal standard solution 30.01 mg.ml⁻¹QC solution of HMX 4.952 mg.ml⁻¹

** = It is suggested that Group 5♀ may have been sampled in error since the dispensary weighing records are correct for the preparation of a nominal 12577 ppm diet

TABLE 28 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
81.94	Standards	236	4.97	17	1	100			
		237	5.03	17	1	100			
		238	5.00	17	1	100			
84.49	QC	239	4.99	17	1	100	85.38	85.49	1.2
		240	4.99	17	1	100	85.25		
		241	5.03	17	1	100	85.85		
33600	63	242	5.04	-	1	117	32233	32925	2.0
		243	4.99	-	1	117	32656		
		244	5.02	-	1	117	33887		

HMX solution 4.82 mg.ml⁻¹Internal standard solution 85.01 mg.ml⁻¹QC solution of HMX 4.97 mg.ml⁻¹

TABLE 29

Analysis of HMX in Diets During 90 Day Toxicity Testing in Rats

Date of Mixing: 22 December 1980 (Week 1)

Date of Analysis: 23 January 1981

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0	13♀	245	5.32	-	-	100	0	0	0
		246	5.45	-	-	100	0		
		247	5.00	-	-	100	0		
1.98	Standards	251	5.18	1	1	100			
		252	4.94	1	1	100			
		253	5.29	1	1	100			
2.56	QC	254	5.16	-	1	100	2.28	2.28	10.9
		255	5.36	1	1	100	2.27		
		256	5.38	1	1	100	2.28		
450	2♂	257	6.72	-	1	100	468	426	5.4
		258	5.15	-	1	100	409		
		259	5.50	-	1	100	400		
431	2♀	263	5.11	-	1	100	412	408	5.3
		264	5.32	-	1	100	404		
		265	5.15	-	1	100	569		

HMX solution 1.98 mg.ml⁻¹Internal standard solution 2.428 mg.ml⁻¹QC solution of HMX 2.56 mg.ml⁻¹

* = Mean of 2 results only

TABLE 29 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
5.66	Standards	269	5.44	1	1	100			
		270	5.30	1	1	100			
		271	5.89	1	1	100			
4.86	QC	272	6.49	1	1	100	5.47	5.50	13.2
		273	6.05	1	1	100	5.53		
		274	5.21	1	1	100	5.51		
1400	3♂	275	4.83	-	1	100	1439	1418	1.3
		276	5.65	-	1	100	1359		
		277	4.92	-	1	100	1457		
1080	3♀	281	5.57	-	1	100	1169	1149	6.4
		282	5.34	-	1	100	1134		
		283	5.10	-	1	100	1143		

HMX solution 5.66 mg.ml⁻¹Internal standard solution 5.776 mg.ml⁻¹QC solution of HMX 4.86 mg.ml⁻¹

TABLE 29 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
11.595	Standards	287	5.00	3	1	100			
		288	5.93	3	1	100			
		289	5.75	3	1	100			
10.86	QC	290	5.66	3	1	100	11.30	11.19	3.0
		291	5.35	3	1	100	11.19		
		292	5.05	3	1	100	11.07		
2916	4♀	293	6.85	-	1	103	2772	2795	4.2
		294	5.25	-	1	103	2788		
		295	5.87	-	1	103	2824		

HMX solution 3.865 mg.ml⁻¹Internal standard solution 11.836 mg.ml⁻¹QC solution of HMX 3.62 mg.ml⁻¹

TABLE 29 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
23.06	Standards	299	5.12	5	1	100			
		300	5.18	5	1	100			
		301	4.82	5	1	100			
20.43	QC	302	4.96	5	1	100	20.47	20.70	1.3
		303	4.96	5	1	100	20.49		
		304	5.04	5	1	100	21.15		
4345	43	305	6.36	-	1	105	4320	4357	0.3
		306	5.17	-	1	105	4392		
		307	6.31	-	1	105	4358		
6526	52	311	5.60	-	1	105	6632	6721	3.0
		312	6.50	-	1	105	6958		
		313	5.30	-	1	105	6573		

HMX solution 4.612 mg.ml⁻¹Internal standard solution 22.812 mg.ml⁻¹QC solution of HMX 4.085 mg.ml⁻¹

TABLE 29 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
31.20	Standards	317	2.46	6	1	100			
		318	2.99	6	1	100			
		319	2.91	6	1	100			
29.30	QC	320	2.94	6	1	100	30.07	29.31	0
		321	2.92	6	1	100	29.41		
		322	2.60	6	1	100	28.44		
12921	5♂	323	3.23	-	1	106	12882	13166	1.9
		324	2.99	-	1	106	13147		
		325	2.42	-	1	106	13470		
15833	6♀	329	2.47	-	1	106	16760	16580	4.7
		330	3.08	-	1	106	16400		
		331	2.26	-	1	106	24167		

HMX solution 5.20 mg.ml⁻¹Internal standard solution 30.072 mg.ml⁻¹QC solution of HMX 4.884 mg.ml⁻¹

* = Mean of 2 results only

TABLE 29 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
88.20	Standards	335	2.87	17	1	100			
		336	2.50	17	1	100			
		337	2.38	17	1	100			
81.311	QC	338	2.90	17	1	100	81.36	82.11	1.0
		339	2.63	17	1	100	83.43		
		340	2.49	17	1	100	81.55		
37538	63	341	2.38	-	1	117	36085	36915	1.7
		342	3.03	-	1	117	37335		
		343	2.46	-	1	117	37325		

HMX solution 5.188 mg.ml⁻¹Internal standard solution 87.608 mg.ml⁻¹QC solution of HMX 4.783 mg.ml⁻¹

TABLE 30

Analysis of HMX In Diets During 90 Day Toxicity Testing In Rats

Date of Mixing: 29 December 1980 (Week 2)

Date of Analysis: 23 January 1981

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0	1♂♀	248	4.97	-	-	100	0	0	0
		249	5.20	-	-	100	0		
		250	5.13	-	-	100	0		
480	2♂	260	4.97	-	1	100	483	482	0.4
		261	5.68	-	1	100	474		
		262	5.10	-	1	100	488		
467	2♂	266	5.37	-	1	100	911	896	•
		267	5.17	-	1	100	866		
		268	5.14	-	1	100	911		
1389	3♂	278	4.74	-	1	100	1453	1494	7.6
		279	5.42	-	1	100	1507		
		280	6.24	-	1	100	1522		

N.B. Analysed with diet prepared 22 December 1980 using the same standard solutions

• = Since the values obtained are markedly different from the nominal concentrations the archive samples for these groups have also been analysed (see p. 65)

TABLE 30 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
1062	3♀	284	5.34	-	1	100	521	548	•
		285	4.88	-	1	100	570		
		286	5.04	-	1	100	552		
2527	4♀	296	5.18	-	1	103	2819	2776	9.9
		297	4.85	-	1	103	2771		
		298	5.80	-	1	103	2739		
4288	4♂	308	4.84	-	1	105	4735	4655	8.6
		309	5.27	-	1	105	4695		
		310	5.81	-	1	105	4536		
5654	5♀	314	4.86	-	1	105	6185	5929	4.6
		315	5.21	-	1	105	6054		
		316	5.28	-	1	105	5547		

• = Since the values obtained are markedly different from the nominal concentrations the archive samples for these groups have also been analysed (see p. 65)

TABLE 30 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
12764	53	326	2.82	-	1	106	13099	12959	1.5
		327	2.60	-	1	106	12813		
		328	2.36	-	1	106	12964		
13065	6♀	332	2.47	-	1	106	12874	13817	5.8
		333	2.45	-	1	106	15603		
		334	2.93	-	1	106	12973		
36500	6♂	344	2.93	-	1	117	31888	34104	6.6
		345	2.51	-	1	117	37009		
		346	2.52	-	1	117	33414		

TABLE 30 (continued)

Analysis of Samples from Archives

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
3.84	Standards	434	4.96	1	1	100			
		435	4.99	1	1	100			
		436	4.94	1	1	100			
467	2♀	437	5.34	-	1	100	1053		φ
		438	5.16	-	1	100	1091	1072	1.0
1062	3♀	439	4.89	-	1	100	504		
		440	5.79	-	1	100	460	482	3.2

HMX solution 3.84 mg.ml⁻¹Internal standard solution 4.01 mg.ml⁻¹

φ = Assumes Group 2♀ and Group 3♀ samples have been interchanged

TABLE 31

Analysis of HMX in Diets During 90 Day Toxicity Testing in Rats

Date of Mixing: 5 January 1981 (Week 3)

Date of Analysis: 21 January 1981

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0	13♀	347	4.88	-	-	100	0	0	0
		348	4.91	-	-	100	0		
		349	5.09	-	-	100	0		
1.68	Standards	350	5.21	1	1	100			
		351	4.90	1	1	100			
		352	4.98	1	1	100			
1.66	QC	353	5.18	1	1	100	1.76	1.82	9.6
		354	5.11	1	1	100	1.85		
		355	5.61	1	1	100	1.84		
506	2♂	356	5.31	-	1	100	552	549	8.5
		357	5.70	-	1	100	543		
		358	5.32	-	1	100	551		
474	2♀	359	5.70	-	1	100	517	533	12.4
		360	5.69	-	1	100	541		
							542		
		361	5.43	-	1	100	539		
							524		

HMX solution 1.68 mg.ml⁻¹Internal standard solution 1.732 mg.ml⁻¹QC solution of HMX 1.66 mg.ml⁻¹

TABLE 31 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
5.00	Standards	362	4.87	1	1	100			
		363	4.79	1	1	100			
		364	5.58	1	1	100			
5.74	QC	365	5.77	1	1	100	5.52	5.28	8.0
		366	5.93	1	1	100	5.09		
		367	5.02	1	1	100	5.24		
1482	33	368	5.52	-	1	100	1459	1449	2.2
		369	5.57	-	1	100	1447		
		370	5.16	-	1	100	1440		
1114	32	371	5.41	-	1	100	1169	1156	3.7
		372	5.71	-	1	100	1171		
		373	5.31	-	1	100	1127		

HMX solution 5.00 mg.ml⁻¹Internal standard solution 4.432 mg.ml⁻¹QC solution of HMX 5.74 mg.ml⁻¹

TABLE 31 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
12.24	Standards	374	4.78	3	1	100			
		375	5.22	3	1	100			
		376	5.45	3	1	100			
9.12	QC	377	5.68	3	1	100	9.02	9.07	0.5
		378	5.19	3	1	100	9.12		
		379	5.41	3	1	100	9.08		
2614	42	380	5.77	-	1	103	2496	2487	4.8
		381	5.37	-	1	103	2387		
		382	4.91	-	1	103	2579		

HMX solution 4.08 mg.ml⁻¹Internal standard solution 11.828 mg.ml⁻¹QC solution of HMX 3.04 mg.ml⁻¹

TABLE 31 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
22.30	Standards	383	5.65	5	1	100			
		384	5.00	5	1	100			
		385	5.00	5	1	100			
15.56	QC	386	5.24	5	1	100	15.28	15.91	2.2
		387	5.91	5	1	100	16.30		
		388	5.61	5	1	100	16.15		
4500	43	389	5.53	-	1	105	4674	4532	0.7
		390	5.89	-	1	105	4195		
		391	4.84	-	1	105	4727		
5850	52	392	5.53	-	1	105	5827	6020	2.9
		393	6.38	-	1	105	6132		
		394	5.71	-	1	105	6100		

HMX solution 4.46 mg.ml⁻¹Internal standard solution 22.18 mg.ml⁻¹QC solution of HMX 5.19 mg.ml⁻¹

TABLE 31 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
31.34	Standards	395	2.59	6	1	100			
		396	2.64	6	1	100			
		397	2.50	6	1	100			
29.52	QC	398	2.48	6	1	100	28.92	29.05	1.7
		399	2.26	6	1	100	28.84		
		400	2.68	6	1	100	29.39		
13331	5♂	401	2.72	-	1	106	14306	13775	3.3
		402	3.19	-	1	106	13337		
		403	3.14	-	1	106	13681		
13893	6♀	404	2.70	-	1	106	13455	13607	2.1
		405	2.53	-	1	106	13950		
		406	2.81	-	1	106	13416		

HMX solution 5.224 mg.ml⁻¹Internal standard solution 30.50 mg.ml⁻¹QC solution of HMX 4.92 mg.ml⁻¹

TABLE 31 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
86.04	Standards	407	2.22	17	1	100			
		408	2.70	17	1	100			
		409	2.40	17	1	100			
85.085	QC	410	2.65	17	1	100	95.78	89.06	4.7
		411	2.46	17	1	100	85.08		
		412	2.18	17	1	100	86.33		
38000	63	413	2.29	-	1	117	37141	36757	3.3
		414	2.37	-	1	117	36315		
		415	2.44	-	1	117	36814		

HMX solution 5.061 mg.ml⁻¹Internal standard solution 81.69 mg.ml⁻¹QC solution of HMX 5.005 mg.ml⁻¹

TABLE 32

Analysis of HMX in Diets During 90 Day Toxicity Testing in Rats

Date of Mixing: 26 January 1981 (Week 6)

Date of Analysis: 5 February 1981

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
2.29	Standards	441	5.34	1	1	50			
		442	5.96	1	1	50			
		443	6.25	1	1	50			
3.86	QC	444	6.61	1	1	50	4.02	4.02	4.1
		445	5.57	1	1	50	4.03		
		446	5.53	1	1	50	4.02		
0	13♀	447	4.68	-	1	50	0	0	0
		448	5.08	-	1	50	0		
		449	5.43	-	1	50	0		
659	2♂	450	5.61	-	1	50	602	607	7.9
		451	6.88	-	1	50	593		
		452	5.59	-	1	50	626		
561	2♀	453	5.50	-	1	50	594	598	6.6
		454	5.94	-	1	50	601		
		455	6.08	-	1	50			

HMX solution 2.29 mg.ml⁻¹Internal standard solution 3.32 mg.ml⁻¹QC solution of HMX 3.86 mg.ml⁻¹

* = Mean of 2 results only

TABLE 32 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
7.94	Standards	456	5.39	2	1	100			
		457	5.38	2	1	100			
		458	5.70	2	1	100			
8.54	QC	459	5.45	2	1	100	8.77	8.85	3.6
		460	5.38	2	1	100	8.81		
		461	5.44	2	1	100	8.98		
1982	33	462	5.00	-	1	102	1783	1811	8.6
		463	4.69	-	1	102	1766		
		464	6.17	-	1	102	1833		
1294	32	465	5.31	-	1	102	1269	1272	1.7
		466	4.96	-	1	102	1245		
		467	5.65	-	1	102	1303		

HMX solution 3.97 mg.ml⁻¹Internal standard solution 9.83 mg.ml⁻¹QC solution of HMX 4.27 mg.ml⁻¹

TABLE 32 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
26.43	Standards	468	6.57	5	1	100			
		469	4.99	5	1	100			
		470	5.07	5	1	100			
27.05	QC	471	4.42	5	1	100	26.98	26.80	0.9
		472	4.94	5	1	100	26.72		
		473	4.95	5	1	100	26.71		
5732	43	474	5.93	-	1	105	5197	5308	7.4
		475	4.71	-	1	105	5458		
		476	4.97	-	1	105	5268		
3076	42	477	5.40	-	1	105	3323	3056	0.7
		478	6.84	-	1	105	2926		
		479	5.18	-	1	105	2919		
7027	52	480	4.90	-	1	105	7041	7011	0.2
		481	5.31	-	1	105	7013		
		482	4.97	-	1	105	6978		

HMX solution 5.285 mg.ml⁻¹Internal standard solution 27.29 mg.ml⁻¹QC solution of HMX 5.41 mg.ml⁻¹

TABLE 32 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
42.82	Standards	483	2.60	10	1	90			
		484	2.86	10	1	90			
		485	2.90	10	1	90			
43.82	QC	486	2.80	10	1	90	45.63	45.10	2.9
		487	2.66	10	1	90	44.84		
		488	2.38	10	1	90	44.82		
17441	5♂	489	3.27	-	1	100	14939	14426	17.3
		490	2.20	-	1	100	14159		
		491	2.51	-	1	100	14179		
16565	6♀	492	3.35	-	1	100	14178	15033	9.2
		493	2.91	-	1	100	15900		
		494	3.23	-	1	100	15020		

HMX solution 4.282 mg.ml⁻¹Internal standard solution 41.9 mg.ml⁻¹QC solution of HMX 4.382 mg.ml⁻¹

+ = Repeated using different standard solution of HMX (see p. 77)

TABLE 32 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
127.28	Standards	495	2.83	25	1	75			
		496	2.22	25	1	75			
		497	2.25	25	1	75			
125.45	QC	498	2.24	25	1	75	125.01	124.71	0.6
		499	2.76	25	1	75	123.91		
		500	3.09	25	1	75	125.20		
49325	63	501	2.15	-	1	100	45065	46616	5.5
		502	2.06	-	1	100	47071		
		503	2.58	-	1	100	47713		

HMX solution 5.09 mg.ml⁻¹Internal standard solution 121.3 mg.ml⁻¹QC solution of HMX 5.02 mg.ml⁻¹

TABLE 32 (continued)

Repeat Analyses

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
50.8	Standards	483R	2.50	10	1	90			
		484R	2.50	10	1	90			
		485R	2.50	10	1	90			
17441	53	489R	3.32	-	1	100	16129	16966	2.7
		490R	3.25	-	1	100	18000		
		491R	3.41	-	1	100	16769		

HMX solution 5.08 mg.ml⁻¹Internal standard solution 48.86 mg.ml⁻¹

TABLE 33

Analysis of HMX In Diets During 90 Day Toxicity Testing In Rats

Date of Mixing: 16 February 1981 (Week 9)

Date of Analysis: 19 February 1981

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
3.23	Standards	504	4.88	1	1	50			
		505	5.88	1	1	50			
		506	4.94	1	1	50			
3.40	QC	507	4.94	1	1	50	3.45	3.47	2.1
		508	5.44	1	1	50	3.49		
		509	5.99	1	1	50	3.47		
0	13♀	510	5.92	-	1	50	0	0	0
		511	4.92	-	1	50	0		
		512	4.76	-	1	50	0		
715	2♂	513	5.12	-	1	50	704	698	2.4
		514	6.00	-	1	50	718		
		515	5.05	-	1	50	672		
606	2♀	516	5.41	-	1	50	651	590	2.6
		517	5.90	-	1	50	513		
		518	5.85	-	1	50	606		

HMX solution 3.23 mg.ml⁻¹Internal standard solution 2.93 mg.ml⁻¹QC solution of HMX 3.40 mg.ml⁻¹

TABLE 33 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
7.86	Standards	519	5.40	2	1	100			
		520	5.32	2	1	100			
		521	5.67	2	1	100			
9.04	QC	522	5.47	2	1	100	8.80	8.87	1.9
		523	6.00	2	1	100	8.93		
		524	5.28	2	1	100	8.88		
2158	33	525	5.72	-	1	100	2107	2115	2.0
		526	5.68	-	1	100	2117		
		527	5.44	-	1	100	2121		
1377	32	528	5.30	-	1	100	1303	1345	2.3
		529	5.02	-	1	100	1343		
		530	6.24	-	1	100	1390		

HMX solution 3.93 mg.ml⁻¹Internal standard solution 8.09 mg.ml⁻¹QC solution of HMX 4.52 mg.ml⁻¹

TABLE 33 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
26.03	Standards	531	4.95	5	1	100			
		532	6.22	5	1	100			
		533	5.21	5	1	100			
30.20	QC	534	5.90	5	1	100	30.41	30.37	0.6
		535	5.31	5	1	100	30.10		
		536	6.00	5	1	100	30.60		
6440	43	537	5.50	-	1	105	6083	6268	2.7
		538	5.28	-	1	105	6574		
		539	5.13	-	1	105	6146		
3348	42	540	5.07	-	1	105	3301	3297	1.5
		541	5.43	-	1	105	3263		
		542	5.57	-	1	105	3326		
7372	52	543	5.42	-	1	105	7292	7295	1.0
		544	5.93	-	1	105	7179		
		545	5.46	-	1	105	7413		

HMX solution 5.205 mg.ml⁻¹Internal standard solution 30.225 mg.ml⁻¹QC solution of HMX 6.04 mg.ml⁻¹

TABLE 33 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
49.16	Standards	546	2.69	10	1	100			
		547	2.45	10	1	100			
		548	2.95	10	1	100			
48.92	QC	549	2.88	10	1	100	48.92	49.00	0.2
		550	2.97	10	1	100	49.68		
		551	2.90	10	1	100	48.41		
19105	53	552	2.61	-	1	110	15202 15754	16582	13.2
		553	2.78	-	1	110	15776 16788		
		554	2.50	-	1	110	17796 18178		
18289	62	555	2.66	-	1	110	17900	17463	4.5
		556	3.16	-	1	110	17367		
		557	2.71	-	1	110	17121		

HMX solution 4.916 mg.ml⁻¹Internal standard solution 47.83 mg.ml⁻¹QC solution of HMX 4.892 mg.ml⁻¹

TABLE 33 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
139.325	Standards	558	3.10	25	1	75			
		559	2.36	25	1	75			
		560	2.44	25	1	75			
137.25	QC	561	2.84	25	1	75	133.72	135.50	1.3
		562	2.61	25	1	75	134.47		
		563	3.50	25	1	75	138.32		
53864	63	564	2.72	-	1	100	57551	52468	2.6
		565	2.50	-	1	100	48764		
		566	2.53	-	1	100	51089		

HMX solution 5.573 mg.ml⁻¹Internal standard solution 133.62 mg.ml⁻¹QC solution of HMX 5.49 mg.ml⁻¹

TABLE 34

Analysis of HMX in Diets During 90 Day Toxicity Testing in Rats

Date of Mixing: 9 March (termination)

Date of Analysis: 20 March 1981

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
3.90	Standards	567	5.06	1	1	50			
		568	5.06	1	1	50			
		569	5.02	1	1	50			
3.44	QC	570	5.15	1	1	50	3.53	3.52	2.3
		571	4.94	1	1	50	3.45		
		572	5.38	1	1	50	3.58		
0	1♂	573	5.20	-	1	50	0	0	0
		574	4.80	-	1	50	0		
		575	4.86	-	1	50	0		
729	2♂	576	6.75	-	1	50	705	716	1.8
		577	5.15	-	1	50	698		
		578	5.89	-	1	50	746		
618	2♀	579	5.21	-	1	50	665	661	7.0
		580	4.53	-	1	50	663		
		581	5.85	-	1	50	655		

HMX solution 3.90 mg.ml⁻¹Internal standard solution 3.86 mg.ml⁻¹QC solution of HMX 3.44 mg.ml⁻¹

TABLE 34 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
10.38	Standards	582	5.00	2	1	100			
		583	5.00	2	1	100			
		584	5.00	2	1	100			
20.40	QC	585	5.00	2	1	100	19.72	19.57	4.1
		586	5.00	2	1	100	19.69		
		587	5.00	2	1	100	19.31		
2214	3♂	588	6.44	-	1	100	2095	2157	2.6
		589	5.68	-	1	100	2178		
		590	5.62	-	1	100	2199		
1373	3♀	591	4.72	-	1	100	1367	1350	2.0
		592	4.52	-	1	100	1303		
		593	5.87	-	1	100	1379		

HMX solution 5.19 mg.ml⁻¹Internal standard solution 10.35 mg.ml⁻¹QC solution of HMX 10.20 mg.ml⁻¹

TABLE 34 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
29.58	Standards	594	5.00	5	1	100			
		595	5.00	5	1	100			
		596	5.00	5	1	100			
31.63	QC	597	5.00	5	1	100	31.58	31.74	0.3
		598	5.00	5	1	100	31.86		
		599	5.00	5	1	100	31.79		
6385	4♂	600	6.32	-	1	105	6356	6437	0.8
		601	5.65	-	1	105	6642		
		602	5.84	-	1	105	6313		
3499	4♀	603	5.82	-	1	105	3550	3553	1.5
		604	5.37	-	1	105	3547		
		605	4.91	-	1	105	3561		
7678	5♀	606	4.81	-	1	105	7714	7617	0.8
		607	5.18	-	1	105	7489		
		608	5.21	-	1	105	7647		

HMX solution 5.915 mg.ml⁻¹Internal standard solution 31.63 mg.ml⁻¹QC solution of HMX 6.325 mg.ml⁻¹

TABLE 34 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
44.9	Standards	609	2.50	10	1	100			
		610	2.50	10	1	100			
		611	2.50	10	1	100			
43.6	QC	612	2.50	10	1	100	43.82	43.89	0.7
		613	2.50	10	1	100	43.87		
		614	2.50	10	1	100	43.98		
18942	5♂	615	2.52	-	1	110	18833	18744	1.0
		616	2.30	-	1	110	17537		
		617	2.46	-	1	110	19863		
17520	6♀	618	2.76	-	1	110	17576	17730	1.2
		619	3.27	-	1	110	17794		
		620	2.36	-	1	110	17819		

HMX solution 4.49 mg.ml⁻¹Internal standard solution 51.27 mg.ml⁻¹QC solution of HMX 4.36 mg.ml⁻¹

TABLE 34 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
142.0	Standards	621	2.27	25	1	75			
		622	2.50	25	1	75			
		623	2.50	25	1	75			
155.4	QC	624	2.50	25	1	75	153.1	153.5	1.2
		625	2.50	25	1	75	154.2		
		626	2.50	25	1	75	153.3		
54105	63	627	2.27	-	1	100	55136	55405	2.4
		628	2.35	-	1	100	55265		
		629	2.71	-	1	100	55815		

HMX solution 5.68 mg.ml⁻¹Internal standard solution 129.0 mg.ml⁻¹QC solution of HMX 6.216 mg.ml⁻¹

TABLE 35

Analysis of HMX in Diets During 90 Day Toxicity Testing in Mice

Date of Mixing: 16 January 1981 (commencement)

Date of Analysis: 18 February 1981

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0.28	Standards	1	5.27	1	1	50			
		2	5.50	1	1	50			
		3	5.54	1	1	50			
0.24	QC	4	4.62	1	1	50	0.36	0.30	24.8
		5	5.43	1	1	50	0.30		
		6	5.12	1	1	50	0.25		
0	1 + 7 32	7	6.94	-	1	50	0	0	0
		8	5.40	-	1	50	0		
		9	5.12	-	1	50	0		
14	23	10	5.00	-	1	50	14	14	0
		11	5.59	-	1	50	14		
		12	5.75	-	1	50	14		

HMX solution 0.28 mg.ml⁻¹Internal standard solution 0.31 mg.ml⁻¹QC solution of HMX 0.24 mg.ml⁻¹

TABLE 35 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
23	2♀	13R	5.93	-	1	50	32	28	21.7
		14R	5.57	-	1	50	28		
		15R	4.99	-	1	50	24		
35	3♂	16	5.76	-	1	50	35	32	8.6
		17	5.47	-	1	50	30		
		18	6.53	-	1	50	30		
68	3♀	19	5.25	-	1	50	61	62	8.8
		20	5.62	-	1	50	62		
		21	5.15	-	1	50	63		
84	4♂	22	5.16	-	1	50	76	79	6.0
		23	6.53	-	1	50	83		
		24	4.67	-	1	50	77		

HMX solution 0.28 mg.ml⁻¹Internal standard solution 0.31 mg.ml⁻¹QC solution of HMX 0.24 mg.ml⁻¹

TABLE 35 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
2.83	Standards	25	5.51	1	1	50			
		26	4.92	1	1	50			
		27	5.46	1	1	50			
2.73	QC	28	4.77	1	1	50	2.88	2.87	5.1
		29	5.49	1	1	50	2.83		
		30	4.87	1	1	50	2.89		
204	42	31	5.40	-	1	50	188	195	4.4
		32	5.22	-	1	50	196		
		33	5.36	-	1	50	202		

HMX solution 2.83 mg.ml⁻¹Internal standard solution 2.06 mg.ml⁻¹QC solution of HMX 2.73 mg.ml⁻¹

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MMX: ANALYSIS OF DOSING FORMULATIONS USED IN ACUTE

2/2

SUB-ACUTE AND SUB-CHRONIC (U) INTRINSIC RESEARCH

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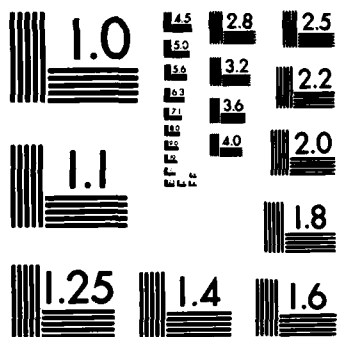
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TABLE 35 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
225	58	34	4.79	-	1	50	222	231	2.7
		35	4.64	-	1	50	223		
		36	5.73	-	1	50	250		
578	59	37	5.01	-	1	50	575	595	2.9
		38	4.90	-	1	50	594		
		39	5.52	-	1	50	616		
662	63	40	5.19	-	1	50	664	657	0.8
		41	4.92	-	1	50	648		
		42	5.18	-	1	50	659		

HMX solution 2.83 mg.ml⁻¹Internal standard solution 2.06 mg.ml⁻¹QC solution of HMX 2.73 mg.ml⁻¹

TABLE 35 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
9.22	Standards	43	4.47	2	1	50			
		44	4.78	2	1	50			
		45	4.82	2	1	50			
11.32	QC	46	5.21	2	1	50	11.75	11.79	4.2
		47	4.55	2	1	50	11.77		
		48	5.54	2	1	50	11.84		
2146	62	49	5.11	-	1	52	2138	2109	1.7
		50	6.14	-	1	52	2063		
		51	5.38	-	1	52	2125		

HMX solution 4.61 mg.ml⁻¹Internal standard solution 22.17 mg.ml⁻¹QC solution of HMX 5.66 mg.ml⁻¹

TABLE 36

Analysis of HMX in Diets During 90 Day Toxicity Testing in Mice

Date of Mixing: 23 January 1981 (Week 1)

Date of Analysis: 28 January 1981

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0.38	Standards	52	5.43	1	1	50			
		53	5.50	1	1	50			
		54	5.14	1	1	50			
0.28	QC	55	5.42	1	1	50	0.28	0.29	3.6
		56	5.89	1	1	50	0.29		
		57	5.97	1	1	50	0.29		
0	1 + 7 ♀	58	5.75	-	1	50	0	0	0
		59	5.39	-	1	50	0		
		60	5.20	-	1	50	0		
16	2♂	61	5.96	-	1	50	15	16	0
		62	4.83	-	1	50	17		
		63	5.52	-	1	50	16		

HMX solution 0.38 mg.ml⁻¹Internal standard solution 0.32 mg.ml⁻¹QC solution of HMX 0.28 mg.ml⁻¹

TABLE 36 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
25	2♀	64	5.17	-	1	50	26	25	0
		65	5.87	-	1	50	27		
		66	4.73	-	1	50	23		
39	3♂	67	5.01	-	1	50	41	39	0
		68	5.26	-	1	50	37		
		69	5.34	-	1	50	38		
78	3♀	70	4.29	-	1	50	80	79	1
		71	4.72	-	1	50	78		
		72	5.30	-	1	50	78		
113	4♂	73	5.07	-	1	50	112	113	0
		74	5.25	-	1	50	114		
		75	4.75	-	1	50	113		

HMX solution 0.38 mg.ml⁻¹Internal standard solution 0.32 mg.ml⁻¹QC solution of HMX 0.28 mg.ml⁻¹

TABLE 36 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
2.71	Standards	76	4.47	1	1	50			
		77	5.52	1	1	50			
		78	4.89	1	1	50			
3.10	QC	79	4.89	1	1	50	3.18	3.19	2.9
		80	5.44	1	1	50	3.17		
		81	5.84	1	1	50	3.21		
201	4♀	82	4.79	-	1	50	208	205	2.0
		83	5.33	-	1	50	204		
		84	5.28	-	1	50	203		

HMX solution 2.71 mg.ml⁻¹Internal standard solution 2.25 mg.ml⁻¹QC solution of HMX 3.10 mg.ml⁻¹

TABLE 36 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
257	5♂	85	4.71	-	1	50	285	274	6.6
		86	5.21	-	1	50	263		
		87	4.94	-	1	50	274		
575	5♀	88	5.16	-	1	50	577	569	1.0
		89	6.88	-	1	50	587		
		90	5.89	-	1	50	543		
688	6♂	91	5.00	-	1	50	716	708	2.9
		92	4.58	-	1	50	701		
		93	4.92	-	1	50	708		

HMX solution 2.71 mg.ml⁻¹Internal standard solution 2.25 mg.ml⁻¹QC solution of HMX 3.10 mg.ml⁻¹

TABLE 36 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
10.56	Standards	94	5.32	2	1	50			
		95	5.80	2	1	50			
		96	6.08	2	1	50			
12.16	QC	97	4.90	2	1	50	12.17	12.24	0.7
		98	5.37	2	1	50	12.32		
		99	5.60	2	1	50	12.24		
1753	6♀	100	5.20	-	1	52	1729	1726	1.5
		101	4.76	-	1	52	1712		
		102	5.38	-	1	52	1736		

HMX solution 5.28 mg.ml⁻¹Internal standard solution 10.36 mg.ml⁻¹QC solution of HMX 6.08 mg.ml⁻¹

TABLE 37

Analysis of HMX in Diets During 90 Day Toxicity Testing in Mice

Date of Mixing: 30 January 1981 (Week 2)

Date of Analysis: 17 February 1981

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0.37	Standards	103	4.75	1	1	50			
		104	5.25	1	1	50			
		105	5.63	1	1	50			
0.32	QC	106	5.49	1	1	50	0.34	0.33	3.1
		107	4.95	1	1	50	0.32		
		108	4.80	1	1	50	0.32		
0	1 + 7 ♀	109	5.55	-	1	50	0	0	0
		110	5.66	-	1	50	0		
		111	5.05	-	1	50	0		
15	2♂	112	5.01	-	1	50	18	Analysis Repeated (see p. 103)	
		113	5.97	-	1	50	39		
		114	4.90	-	1	50	18		

HMX solution 0.37 mg.ml⁻¹Internal standard solution 0.37 mg.ml⁻¹QC solution of HMX 0.32 mg.ml⁻¹

TABLE 37 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
26	2♀	115	6.86	-	1	50	26	28	7.7
		116	5.20	-	1	50	31		
		117	6.19	-	1	50	28		
38	3♂	118	5.47	-	1	50	38	39	2.6
		119	5.46	-	1	50	39		
		120	4.44	-	1	50	40		
90	3♀	121	5.71	-	1	50	96	92	2.2
		122	5.10	-	1	50	86		
		123	5.36	-	1	50	93		
105	4♂	124	6.14	-	1	50	103	102	2.9
		125	4.76	-	1	50	100		
		126	5.27	-	1	50	103		

HMX solution 0.37 mg.ml⁻¹Internal standard solution 0.37 mg.ml⁻¹QC solution of HMX 0.32 mg.ml⁻¹

TABLE 37 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
2.48	Standards	127	4.90	1	1	50			
		128	4.87	1	1	50			
		129	4.82	1	1	50			
2.76	QC	130	5.10	1	1	50	2.73	2.78	0.7
		131	4.87	1	1	50	2.80		
		132	5.65	1	1	50	2.80		
258	4♀	133	5.24	-	1	50	249	257	0.4
		134	5.49	-	1	50	271		
		135	4.79	-	1	50	252		

HMX solution 2.48 mg.ml⁻¹Internal standard solution 2.57 mg.ml⁻¹QC solution of HMX 2.76 mg.ml⁻¹

TABLE 37 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
280	5♂	136	5.34	-	1	50	277	276	1.4
		137	4.85	-	1	50	278		
		138	4.92	-	1	50	273		
754	5♀	139	5.63	-	1	50	744	750	0.5
		140	5.48	-	1	50	742		
		141	5.11	-	1	50	764		
756	6♂	142	4.80	-	1	50	767	754	0.5
		143	6.30	-	1	50	761		
		144	6.05	-	1	50	735		

HMX solution 2.48 mg.ml⁻¹Internal standard solution 2.57 mg.ml⁻¹QC solution of HMX 2.76 mg.ml⁻¹

TABLE 37 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
11.70	Standards	145	5.92	3	1	50			
		146	4.56	3	1	50			
		147	5.96	3	1	50			
15.09	QC	148	6.38	3	1	50	15.06	14.89	1.3
		149	5.61	3	1	50	14.50		
		150	5.70	3	1	50	15.12		
2650	6♀	151	5.39	-	1	53	2264	Analysis Repeated (see p. 103)	
		152	4.73	-	1	53	2354		
		153	5.75	-	1	53	2434		

HMX solution 3.90 mg.ml⁻¹Internal standard solution 12.05 mg.ml⁻¹QC solution of HMX 5.03 mg.ml⁻¹

TABLE 37 (continued)

Repeat Analyses

Nominal Value	Group	Analytical Sample No.	Value Found	Mean Value Found	Deviation of Mean Value (%)
15	28	112R	14	14	6.7
		113R	15		
		114R	14		
2650	62	151R	2884	2781	4.9
		152R	2811		
		153R	2647		

TABLE 38

Analysis of HMX in Diets During 90 Day Toxicity Testing in Mice

Date of Mixing: 6 February 1981 (Week 3)

Date of Analysis: 17 February 1981

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0.125	Standards	154	5.27	1	1	50			
		155	6.38	1	1	50			
		156	5.38	1	1	50			
0.130	QC	157	5.38	1	1	50	0.125	0.126	3.1
		158	5.43	1	1	50	0.127		
		159	5.32	1	1	50	0.126		
0	1 + 7 ♂♀	160	7.02	-	1	50	0	0	0
		161	5.08	-	1	50	0		
		162	5.05	-	1	50	0		

HMX solution 0.125 mg.ml⁻¹Internal standard solution 0.17 mg.ml⁻¹QC solution of HMX 0.130 mg.ml⁻¹

TABLE 38 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
16	28	163	5.55	-	1	50	16	17	6.3
		164	5.23	-	1	50	18		
		165	5.16	-	1	50	16		
27	29	166	5.14	-	1	50	26	25	7.4
		167	5.38	-	1	50	26		
		168	5.90	-	1	50	23		
37	38	169	5.50	-	1	50	37	37	0
		170	6.23	-	1	50	38		
		171	5.33	-	1	50	35		

HMX solution 0.125 mg.ml⁻¹Internal standard solution 0.17 mg.ml⁻¹QC solution of HMX 0.130 mg.ml⁻¹

TABLE 38 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0.48	Standards	172	6.67	1	1	50			
		173	5.33	1	1	50			
		174	6.10	1	1	50			
0.51	QC	175	5.70	1	1	50	0.548	0.546	7.1
		176	5.41	1	1	50	0.548		
		177	5.15	1	1	50	0.543		
85	32	178	6.16	-	1	50	92	87	2.3
		179	5.64	-	1	50	85		
		180	5.44	-	1	50	84		
102	48	181	5.91	-	1	50	110	111	8.8
		182	6.32	-	1	50	111		
		183	5.26	-	1	50	111		

HMX solution 0.48 mg.ml⁻¹Internal standard solution 0.46 mg.ml⁻¹QC solution of HMX 0.51 mg.ml⁻¹

TABLE 38 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
1.46	Standards	184	5.12	1	1	50			
		185	5.93	1	1	50			
		186	6.55	1	1	50			
1.650	QC	187	5.44	1	1	50	1.669	1.684	2.1
		188	6.17	1	1	50	1.675		
		189	6.82	1	1	50	1.707		
247	4♀	190	5.26	-	1	50	237	243	1.6
		191	5.23	-	1	50	247		
		192	5.40	-	1	50	245		
282	5♂	193	5.69	-	1	50	267	270	4.3
		194	6.04	-	1	50	263		
		195	5.16	-	1	50	281		

HMX solution 1.46 mg.ml⁻¹Internal standard solution 1.60 mg.ml⁻¹QC solution of HMX 1.650 mg.ml⁻¹

TABLE 38 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
3.67	Standards	196	5.89	1	1	50			
		197	5.21	1	1	50			
		198	5.68	1	1	50			
3.630	QC	199	4.99	1	1	50	3.614	3.625	0.1
		200	5.80	1	1	50	3.576		
		201	5.64	1	1	50	3.684		
642	5♀	202	5.73	-	1	50	635	622	3.1
		203	5.12	-	1	50	641		
		204	5.14	-	1	50	591		
747	6♂	205	5.62	-	1	50	730	719	3.7
		206	5.80	-	1	50	708		
		207	5.27	-	1	50	718		

HMX solution 3.67 mg.ml⁻¹Internal standard solution 3.46 mg.ml⁻¹QC solution of HMX 3.630 mg.ml⁻¹

TABLE 38 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
5.54	Standards	208	5.19	2	1	50			
		209	5.81	2	1	50			
		210	6.07	2	1	50			
12.68	QC	211	5.61	2	1	50	12.68	12.63	0.4
		212	5.57	2	1	50	12.65		
		213	6.54	2	1	50	12.56		
2228	6♀	214	5.30	-	1	52	2258	2279	2.3
		215	5.13	-	1	52	2394		
		216	5.09	-	1	52	2185		

HMX solution 5.54 mg.ml⁻¹Internal standard solution 12.355 mg.ml⁻¹QC solution of HMX 6.34 mg.ml⁻¹

TABLE 39

Analysis of HMX in Diets During 90 Day Toxicity Testing in Mice

Date of Mixing: 27 February 1981 (Week 6)

Date of Analysis: 11 March 1981

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0.174	Standards	217	5.00	1	1	50			
		218	5.39	1	1	50			
		219	5.19	1	1	50			
0.115	QC	220	5.09	1	1	50	0.130	0.129	12.2
		221	4.97	1	1	50	0.129		
		222	5.34	1	1	50	0.127		
0	1 + 7 ♂♀	223	5.00	-	1	50	0	0	0
		224	5.15	-	1	50	0		
		225	5.26	-	1	50	0		

HMX solution 0.174 mg.ml⁻¹Internal standard solution 0.165 mg.ml⁻¹QC solution of HMX 0.115 mg.ml⁻¹

TABLE 39 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
13	28	226	5.18	-	1	50	14	13	0
		227	5.00	-	1	50	13		
		228	5.18	-	1	50	12		
24	29	229	5.07	-	1	50	24	27	12.5
		230	5.03	-	1	50	26		
		231	5.04	-	1	50	31		
31	33	232	5.08	-	1	50	36	35	12.9
		233	5.28	-	1	50	34		
		234	5.08	-	1	50	35		

HMX solution 0.174 mg.ml⁻¹Internal standard solution 0.165 mg.ml⁻¹QC solution of HMX 0.115 mg.ml⁻¹

TABLE 39 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0.506	Standards	235	4.99	1	1	50			
		236	5.00	1	1	50			
		237	5.09	1	1	50			
0.630	QC	238	5.03	1	1	50	0.639	0.636	1.0
		239	5.39	1	1	50	0.628		
		240	5.00	1	1	50	0.641		
70	32	241	5.03	-	1	50	66	70	0
		242	5.07	-	1	50	76		
		243	5.17	-	1	50	68		
76	43	244	5.01	-	1	50	81	78	2.6
		245	5.08	-	1	50	77		
		246	5.14	-	1	50	75		

HMX solution 0.506 mg.ml⁻¹Internal standard solution 0.51 mg.ml⁻¹QC solution of HMX 0.630 mg.ml⁻¹

TABLE 39 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
1.260	Standards	247	5.00	1	1	50			
		248	5.09	1	1	50			
		249	4.99	1	1	50			
1.470	QC	250	5.15	1	1	50	1.413	1.429	2.8
		251	5.13	1	1	50	1.444		
		252	5.25	1	1	50	1.431		
204	4♀	253	5.23	-	1	50	220	208	2.0
		254	5.26	-	1	50	197		
		255	5.13	-	1	50	206		
248	5♂	256	5.03	-	1	50	247	250	0.8
		257	5.04	-	1	50	253		
		258	5.00	-	1	50	250		

HMX solution 1.260 mg.ml⁻¹Internal standard solution 1.495 mg.ml⁻¹QC solution of HMX 1.470 mg.ml⁻¹

TABLE 39 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
3.90	Standards	259	5.06	1	1	50			
		260	5.06	1	1	50			
		261	5.02	1	1	50			
3.44	QC	262	5.15	1	1	50	3.43	3.46	0.6
		263	4.94	1	1	50	3.45		
		264	5.38	1	1	50	3.51		
613	5♀	265	5.50	-	1	50	602	624	1.8
		266	5.23	-	1	50	647		
		267	5.04	-	1	50	622		
645	6♂	268	5.00	-	1	50	639	623	3.4
		269	5.14	-	1	50	621		
		270	5.36	-	1	50	608		

HMX solution 3.90 mg.ml⁻¹Internal standard solution 3.68 mg.ml⁻¹QC solution of HMX 3.44 mg.ml⁻¹

TABLE 39 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
12.56	Standards	271	5.00	2	1	50			
		272	4.99	2	1	50			
		273	5.44	2	1	50			
13.06	QC	274	5.36	2	1	50	13.57	13.35	2.2
		275	5.00	2	1	50	13.20		
		276	5.02	2	1	50	13.29		
1789	6♀	277	5.06	-	1	52	1794	1788	0
		278	5.87	-	1	52	1783		
		279	5.34	-	1	52	1786		

HMX solution 6.28 mg.ml⁻¹Internal standard solution 12.33 mg.ml⁻¹QC solution of HMX 6.53 mg.ml⁻¹

TABLE 40

Analysis of HMX in Diets During 90 Day Toxicity Testing in Mice

Date of Mixing: 20 March 1981 (Week 9)

Date of Analysis: 30 March 1981

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0.134	Standards	280	5.60	1	1	50			
		281	5.62	1	1	50			
		282	5.55	1	1	50			
0.140	QC	283	5.31	1	1	50	0.138	0.141	0.7
		284	5.34	1	1	50	0.143		
		285	5.02	1	1	50	0.143		
0	1 + 7 ♂♀	286	5.12	-	1	50	0	0	0
		287	5.14	-	1	50	0		
		288	5.13	-	1	50	0		

HMX solution 0.134 mg.ml⁻¹Internal standard solution 0.126 mg.ml⁻¹QC solution of HMX 0.140 mg.ml⁻¹

TABLE 40 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
15	28	289	5.75	-	1	50	17	18	20.0
		290	6.08	-	1	50	19		
		291	4.75	-	1	50	17		
27	29	292	5.03	-	1	50	27	26	3.7
		293	5.08	-	1	50	25		
		294	6.35	-	1	50	27		
35	30	295	5.51	-	1	50	37	37	5.7
		296	5.49	-	1	50	36		
		297	5.36	-	1	50	37		

HMX solution 0.134 mg.ml⁻¹Internal standard solution 0.126 mg.ml⁻¹QC solution of HMX 0.140 mg.ml⁻¹

TABLE 40 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0.518	Standards	298	5.02	1	1	50			
		299	5.49	1	1	50			
		300	5.49	1	1	50			
0.398	QC	301	5.74	1	1	50	0.395	0.397	0.3
		302	4.78	1	1	50	0.401		
		303	5.04	1	1	50	0.394		
79	3♀	304	5.42	-	1	50	84	84	6.3
		305	4.42	-	1	50	84		
		306	5.67	-	1	50	83		
89	4♂	307	5.13	-	1	50	96	94	5.6
		308	5.00	-	1	50	96		
		309	5.10	-	1	50	91		

HMX solution 0.518 mg.ml⁻¹Internal standard solution 0.47 mg.ml⁻¹QC solution of HMX 0.398 mg.ml⁻¹

TABLE 40 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
1.46	Standards	310	4.93	1	1	50			
		311	5.66	1	1	50			
		312	5.58	1	1	50			
1.22	QC	313	5.96	1	1	50	1.14	1.19	2.5
		314	5.55	1	1	50	1.21		
		315	4.86	1	1	50	1.21		
238	42	316	4.56	-	1	50	237	231	2.9
		317	5.23	-	1	50	236		
		318	5.48	-	1	50	220		
254	53	319	4.88	-	1	50	290	273	7.5
		320	5.33	-	1	50	259		
		321	5.28	-	1	50	270		

HMX solution 1.46 mg.ml⁻¹Internal standard solution 1.36 mg.ml⁻¹QC solution of HMX 1.22 mg.ml⁻¹

TABLE 40 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
4.35	Standards	322	6.23	1	1	50			
		323	6.82	1	1	50			
		324	6.09	1	1	50			
3.21	QC	325	6.16	1	1	50	3.25	3.24	0.9
		326	5.08	1	1	50	3.27		
		327	5.77	1	1	50	3.20		
685	5♀	328	5.64	-	1	50	696	693	1.2
		329	5.62	-	1	50	698		
		330	5.29	-	1	50	685		
661	6♂	331	4.47	-	1	50	678	679	2.7
		332	5.45	-	1	50	673		
		333	4.05	-	1	50	685		

HMX solution 4.35 mg.ml⁻¹Internal standard solution 3.615 mg.ml⁻¹QC solution of HMX 3.21 mg.ml⁻¹

TABLE 40 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
10.78	Standards	334	5.34	2	1	50			
		335	5.34	2	1	50			
		336	5.39	2	1	50			
11.34	QC	337	4.68	2	1	50	11.22	11.24	0.9
		338	4.82	2	1	50	11.15		
		339	4.76	2	1	50	11.34		
2204	6♀	340	5.04	-	1	52	2240	2190	0.6
		341	4.79	-	1	52	2134		
		342	6.34	-	1	52	2197		

HMX solution 5.39 mg.ml⁻¹Internal standard solution 11.55 mg.ml⁻¹QC solution of HMX 5.67 mg.ml⁻¹

TABLE 41

Analysis of HMX in Diets During 90 Day Toxicity Testing in Mice

Date of Mixing: 10 April 1981 (termination)

Date of Analysis: 24 April 1981

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0.128	Standards	343	4.95	1	1	50			
		344	6.00	1	1	50			
		345	5.66	1	1	50			
0.137	QC	346	5.43	1	1	50	0.133	0.128	6.6
		347	5.40	1	1	50	0.125		
		348	4.60	1	1	50	0.127		
0	1 + 7 ♂♀	349	5.47	-	1	50	0	0	0
		350	7.16	-	1	50	0		
		351	4.77	-	1	50	0		

HMX solution 0.128 mg.ml⁻¹Internal standard solution 0.113 mg.ml⁻¹QC solution of HMX 0.137 mg.ml⁻¹

TABLE 41 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
16	2♂	352	7.83	-	1	50	16	16	0
		353	5.32	-	1	50	16		
		354	5.80	-	1	50	15		
27	2♀	355	6.14	-	1	50	36 35 36	31	14.8
		356	4.70	-	1	50	24 35 24		
		357	6.04	-	1	50	28 28		
41	3♂	358	5.24	-	1	50	38	38	7.3
		359	4.82	-	1	50	38		
		360	5.34	-	1	50	37		

HMX solution 0.128 mg.ml⁻¹Internal standard solution 0.113 mg.ml⁻¹QC solution of HMX 0.137 mg.ml⁻¹++ = Analysis repeated since original values were outwith ± 5 ppm

TABLE 41 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
0.546	Standards	361	4.79	1	1	50			
		362	5.42	1	1	50			
		363	5.18	1	1	50			
0.408	QC	364	5.99	1	1	50	0.356	0.410	0.5
		365	6.12	1	1	50	0.428		
		366	5.22	1	1	50	0.445		
82	39	367	5.46	-	1	50	89 78	82	0
		368	5.06	-	1	50	97 75		
		369	5.86	-	1	50	95 92		
105	43	370	6.79	-	1	50	125 122	109	3.8
		371	6.32	-	1	50	130 105		
		372	5.63	-	1	50	114 101		

HMX solution 0.546 mg.ml⁻¹Internal standard solution 0.570 mg.ml⁻¹QC solution of HMX 0.408 mg.ml⁻¹

++ = Analysis repeated since original values were outwith 10%

Mean concentrations are those from the repeat analyses

TABLE 41 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
1.18	Standards	373	5.23	1	1	50			
		374	5.17	1	1	50			
		375	5.14	1	1	50			
1.03	QC	376	5.27	1	1	50	1.05	1.05	1.9
		377	4.65	1	1	50	1.11		
		378	5.43	1	1	50	0.99		
245	4♀	379	5.91	-	1	50	240	236	3.7
		380	7.05	-	1	50	235		
		381	6.08	-	1	50	232		
302	5♂	382	5.68	-	1	50	303	301	0.3
		383	6.20	-	1	50	301		
		384	5.50	-	1	50	298		

HMX solution 1.18 mg.ml⁻¹Internal standard solution 1.585 mg.ml⁻¹QC solution of HMX 1.03 mg.ml⁻¹

TABLE 41 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
4.60	Standards	385	5.67	1	1	50			
		386	6.05	1	1	50			
		387	5.49	1	1	50			
3.26	QC	388	6.06	1	1	50	3.35	3.41	4.6
		389	5.80	1	1	50	3.49		
		390	5.80	1	1	50	3.39		
728	52	391	5.40	-	1	50	755	749	2.9
		392	5.16	-	1	50	750		
		393	5.36	-	1	50	743		
830	63	394	5.25	-	1	50	871	874	5.3
		395	6.15	-	1	50	869		
		396	5.63	-	1	50	881		

HMX solution 4.60 mg.ml⁻¹Internal standard solution 3.47 mg.ml⁻¹QC solution of HMX 3.26 mg.ml⁻¹

TABLE 41 (continued)

Nominal Value	Group	Analytical Sample No.	Weight Diet Used (g)	Volume HMX Solution Added (ml)	Volume Internal Standard Solution (ml)	Volume Extracting Solvent (ml)	Value Found	Mean Value Found	Deviation of Mean Value (%)
11.14	Standards	397	6.34	2	1	50			
		398	7.34	2	1	50			
		399	5.21	2	1	50			
11.34	QC	400	5.38	2	1	50	11.26	11.27	0.6
		401	5.03	2	1	50	11.24		
		402	5.92	2	1	50	11.30		
2550	67	403	5.33	-	1	52	2494	2531	0.7
		404	5.58	-	1	52	2520		
		405	6.05	-	1	52	2578		

HMX solution 5.57 mg.ml⁻¹Internal standard solution 12.04 mg.ml⁻¹QC solution of HMX 5.67 mg.ml⁻¹

FIGURE 1
Ultraviolet Spectrum of HMX

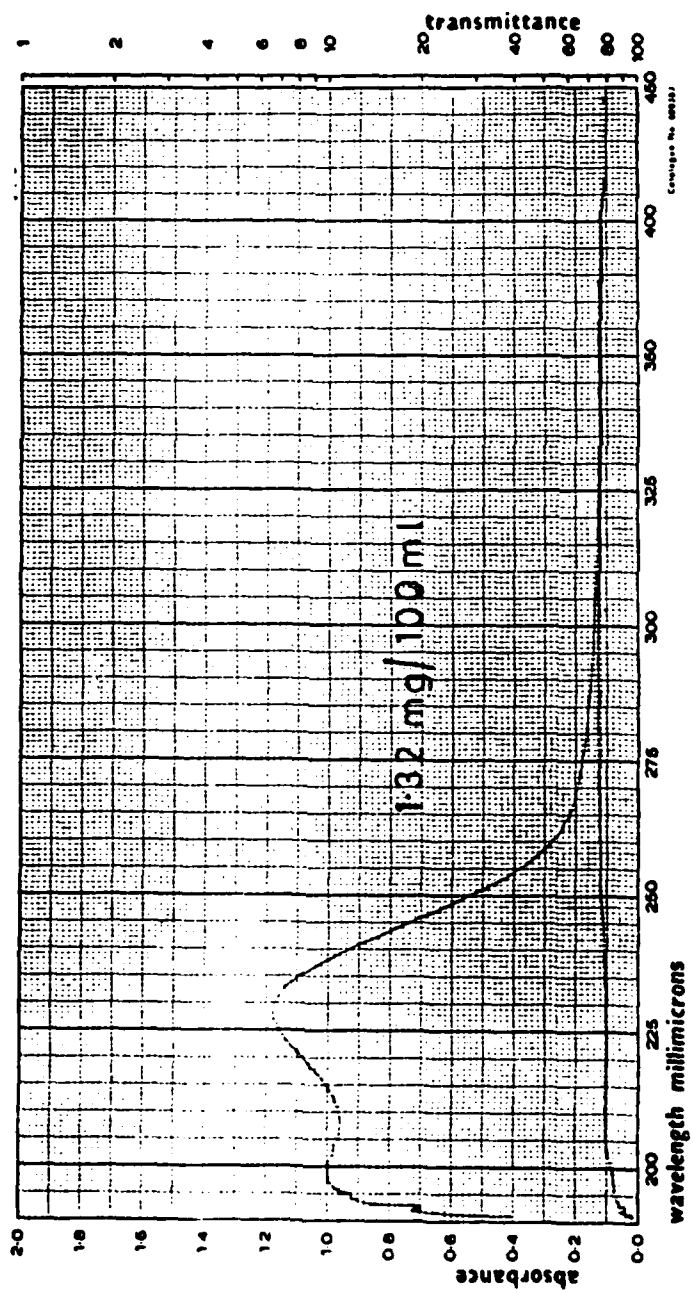


FIGURE 2

HPLC of HMX and Related Explosives (Solvent 1)

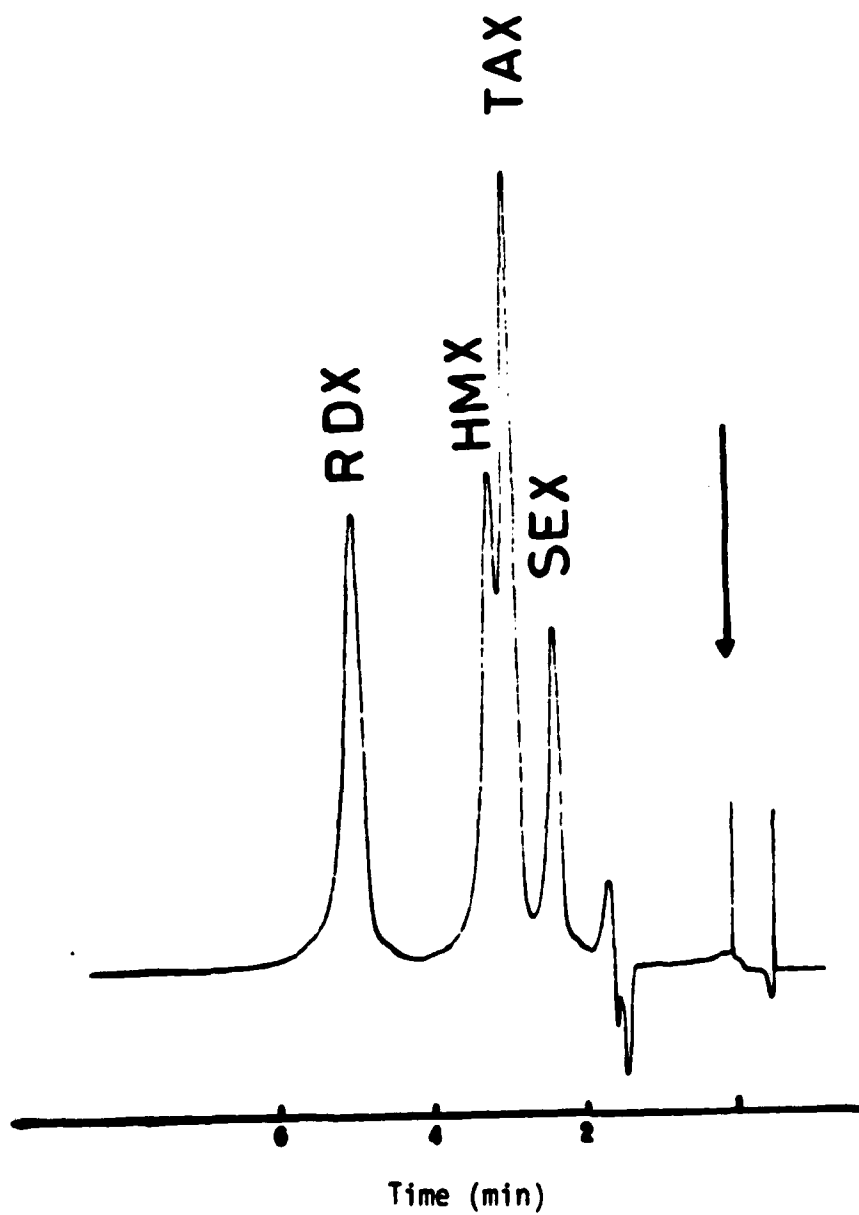


FIGURE 3

HPLC of HMX and RDX (Solvent 2)

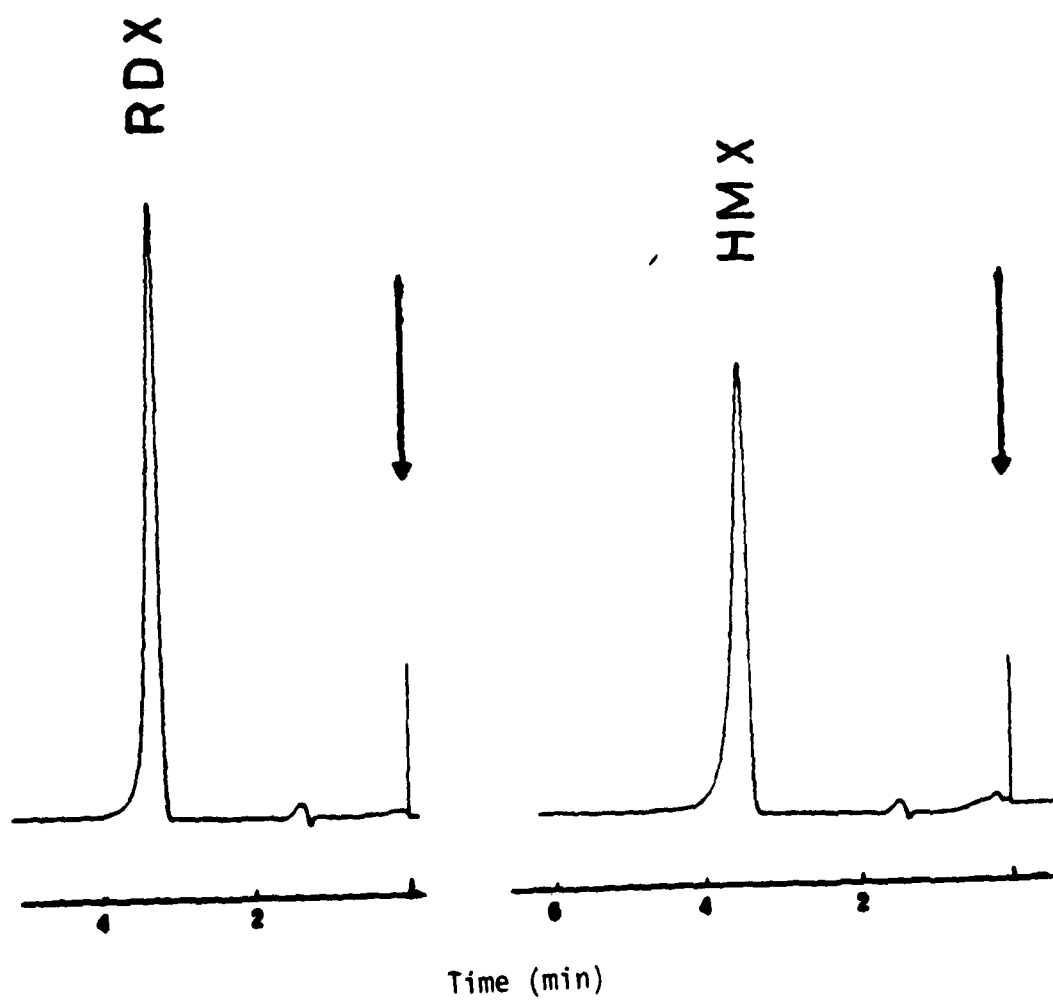


FIGURE 4

HPLC of HMX and RDX (Solvent 2)

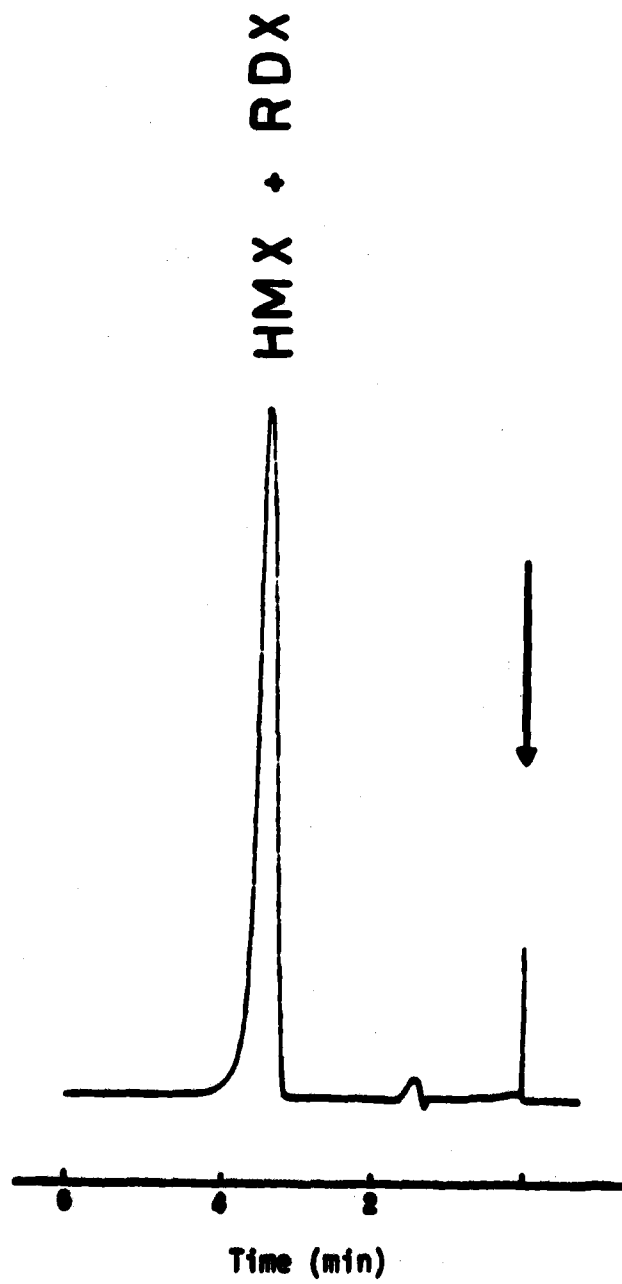


FIGURE 5

HPLC of HMX and Related Explosives (Solvent 2)

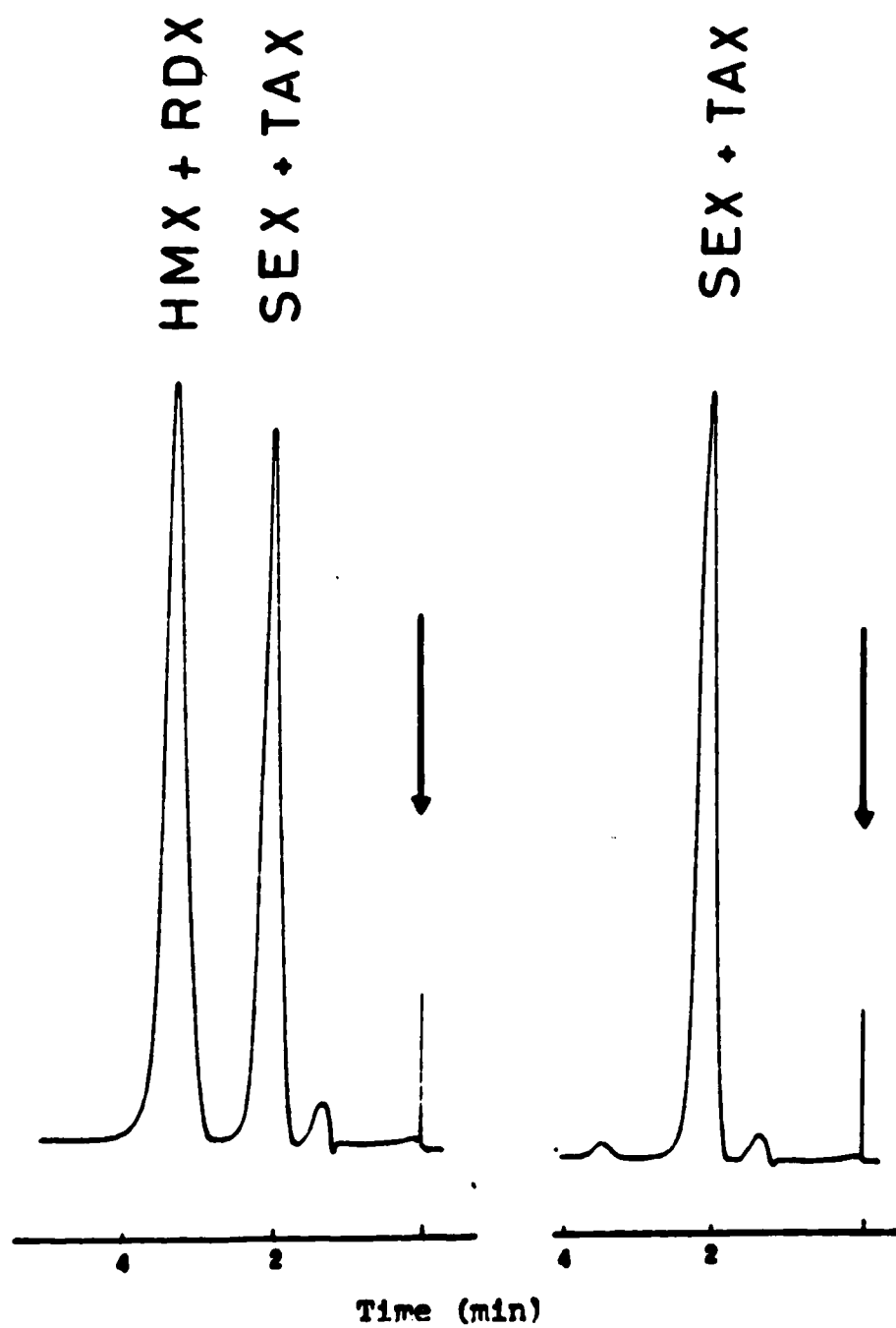


FIGURE 6

HPLC of HMX and Tetryl (Solvent 2)

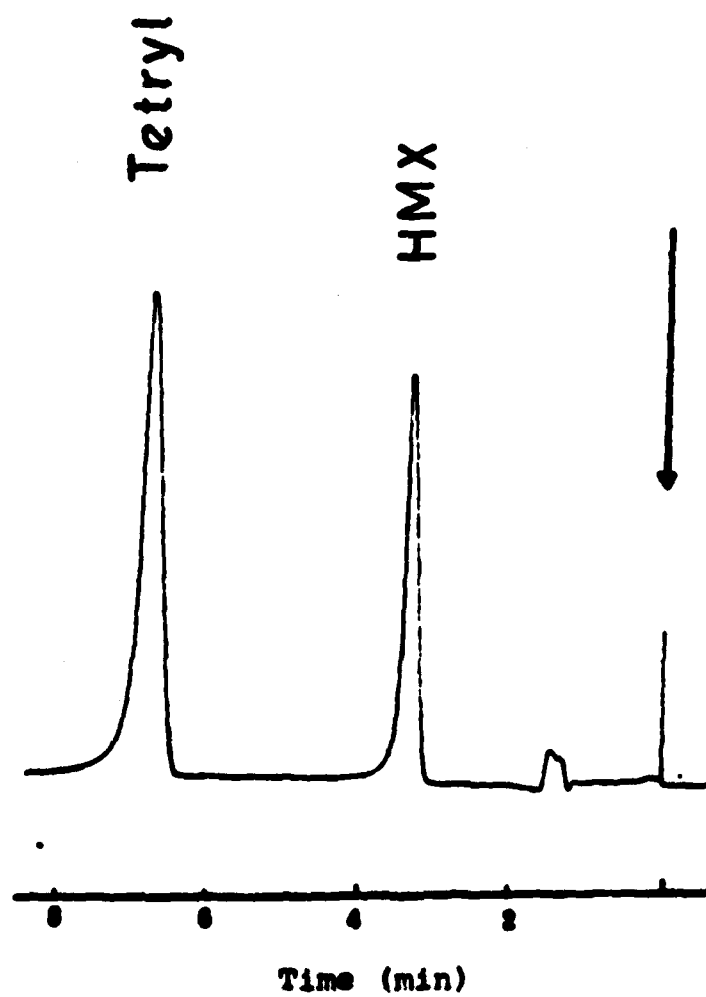


FIGURE 7

HPLC of HMX and 1,3-Dinitrobenzene (DNB)

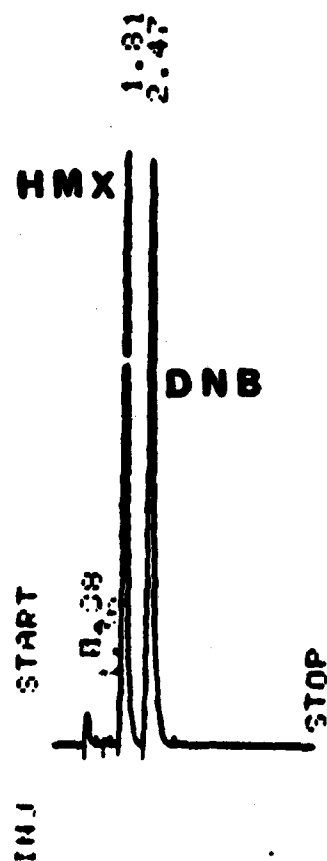
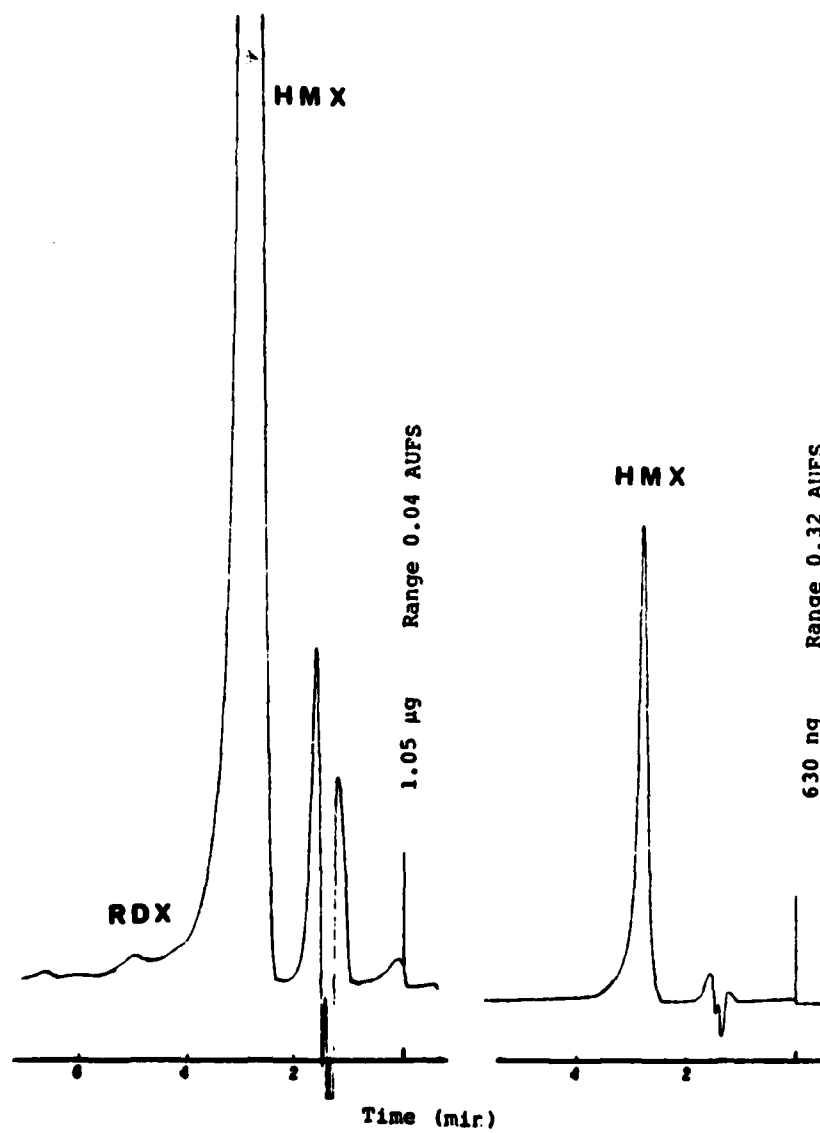


FIGURE 8
HPLC of HMX Solutions (Solvent 1)



APPENDIX 1

**HMX: Analysis of Dosing Formulations Used in Acute, Sub-acute
and Sub-chronic Toxicity Studies
Diet Analysis**

**B.P. NUTRITION (U.K.) LTD.
SPECIAL QUALITY CONTROL OF LABORATORY ANIMAL DIETS**

CERTIFICATE OF ANALYSIS

PRODUCT: RAT & MOUSE NO.1 (MODIFIED) EXPANDED FINE GROUND

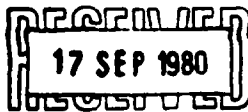
BATCH NO: 919

PREMIX BATCH NO: P110

DATE OF MANUFACTURE: 15TH AUGUST 1980

Nutrient	Found Analysis	Contaminant	Found Analysis	Limit of Detection
Moisture	7.1 %	Fluoride	7.6 mg/kg	10.0 mg/kg
Crude Fat	3.5° %	Nitrate as NaNO ₃	11.0 mg/kg	1.0 mg/kg
Crude Protein	14.9 %	Nitrite as NaNO ₂	< 1.0 mg/kg	1.0 mg/kg
Crude Fibre	2.2 %	Lead	< 1.0 mg/kg	1.0 mg/kg
Ash	4.8 %	Arsenic	0.23 mg/kg	0.2 mg/kg
Calcium	0.69 %	Cadmium	0.13 mg/kg	0.2 mg/kg
Phosphorus	0.53 %	Mercury	< 0.01 mg/kg	0.01 mg/kg
Sodium	0.22 %	Selenium	0.12 mg/kg	0.02 mg/kg
Chlorine	0.34 %			
Potassium	1.10 %			
Magnesium	0.13 %	Total Aflatoxins	NONE DETECTED ug/kg	1 ug/kg each of B1, B2, G1, G2
Iron	231 mg/kg			
Copper	7 mg/kg			
Manganese	55 mg/kg	Total P.C.B.	NONE DETECTED mg/kg	0.001 mg/kg
Zinc	40 mg/kg	Total D.D.T.	0.003 mg/kg	0.001 mg/kg
		Dieldrin	0.001 mg/kg	0.001 mg/kg
		Lindane	0.005 mg/kg	0.001 mg/kg
		Heptachlor	NONE DETECTED mg/kg	0.001 mg/kg
		Malathion	NONE DETECTED mg/kg	0.02 mg/kg
Vitamin A	7000 iu/kg	Total Viable Organisms	1.13×10^3 per gram	1000g
Vitamin E	60 mg/kg	Mesophilic Spores	17.5×10^2 per gram	100g
Vitamin C	mg/kg	Salmonellae Species	NONE DETECTED per gram	Absent in 20 gram
		Presumptive E. Coli	NONE DETECTED per gram	Absent in 10 gram
		E. Coli Type 1	NONE DETECTED per gram	Absent in 10 gram
		Fungal Units	NONE DETECTED per gram	Absent in 10 gram
		Antibiotic Activity		

*repeat 3,4



Signed

C. R. Popplestone

Dated

10/2 September 1980

C. R. POPPLESTONE M.Sc., Ph.D., C.Chem., M.R.I.C.
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PERSONNEL INVOLVED IN PROJECTS 415669 AND 416877

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Department Manager: (Analytical Chemistry)	J.D. Gilbert, B.Sc., Ph.D. C.Chem., F.R.S.C.
Senior Analyst:	M.S. Henderson, B.Sc., Ph.D.
Analysts:	D.W. Carmichael, B.Sc. S. Souter
Project Leader (Acute Toxicology):	S.M.A. Carr
Test Substance Formulator:	A. Soden
Dispensary Assistant:	A. Trench
Quality Assurance:	E.M. Baxendine, B.Sc., A.W. Waddell, B.Sc., Ph.D. F.M. Cunningham, M.Sc., M.I.Biol.

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